EFFECT OF DIFFERENT BONDING AGENTS ON SINTERING BEHAVIOUR OF Al_2O_3-SiO_2 BASED MORTAR

A thesis submitted in partial fulfilment of the requirement for the degree of

MASTER OF TECHNOLOGY
In
Ceramic Engineering
By

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Under the Guidance
Of
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Department Of Ceramic Engineering
National Institute Of Technology
Rourkela, May 2013

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CERTIFICATE

This is to certify that the thesis entitled **"Effect of Different Bonding Agents on sintering behaviour of Al_2O_3-SiO_2 based Mortar"** submitted by Miss. Swagatika Mishra (212CR2502) in partial fulfillment of the requirement for the award of the degree of Master of technology in Ceramic Engineering at National Institute of Technology, Rourkela *is a* record of bonafide research work carried out by her under our supervision and guidance. Her thesis, in our opinion, is worthy of consideration for the award of degree of Master of Technology in accordance with the regulations of the institute.

To the best of our knowledge, no part of this thesis has been submitted to any other University or Institution for the award of a degree.

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ABSTRACT

Refractory Mortar is a mixture of fine particles which are used in the form of a paste to bind refractory bricks after the addition of water to the mixture. The composition and chemistry of the mortar need to be similar to that of the brick to be joined by the mortar. The service life of a refractory lining not only depends on the properties bricks but also on joints made by mortar. More precisely to say mortar joints in a refractory walls is the weakest part of the wall. Mortars used to bind the bricks are generally low cost materials as compared to bricks. But its importance is much more than brick. Because it forms the lining between the refractory bricks; if failure occurs, it causes the damage of the whole refractory lining.

Mortar consists of three major components namely aggregate, binder and water. The composition of aggregates needs to be same as the brick composition but the particle size and its distribution are different for both the cases. Particle size of the aggregates used in the mortar composition needs to be less than 200 meshes so that it will give a paste like structure when water is added to it. After the aggregate content the major constituent of mortar is the binder. Bonding system of mortar strongly depends upon the nature of the binder and the water content to give a required consistency. So a suitable binder must be chosen which provides excellent green as well as fired strength to a mortar.

In the present study, attempts have been made to develop 45% Al_2O_3 mortar to join 45-50% alumina bricks with suitable binder. Chinese bauxite, fire clay grog, and sillimanite sand have been used as aggregates to prepare the mortar. Three binders chosen are bentonite, plastic clay and sodium silicate liquid. Attempt has been made to study the effect of binders on the physical and mechanical properties of the mortar thus developed.

Analysis has been done by considering all the physical and mechanical properties of the mortar found by using different binders in different percentage. It has been found by using bentonite as a binder that, all the mechanical properties of the mortar increases with increase in bentonite content also with increase in temperature. It is also concluded that by using plastic clay as a binding agent, mechanical also physical behaviour of mortar increases with increase in plastic clay content. By using sodium silicate liquid as a binder all the mechanical properties of the mortar specially bond strength of mortar increases drastically with increase in temperature also with sodium silicate liquid content as compared to other binders studied. Among all binder studied, sodium silicate liquid was found to be better than other.

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Chapter 1

Introduction

Bricks and mortar share integral parts of a refractory wall lining. In conventional refractory lining bricks are joined together by a suitable mortar. The composition and chemistry of the mortar are similar to that of the brick to be joined by the mortar. The life of the refractory lining not only depends on the bricks but also on the corrosion erosion behaviour of the joints made up of mortar. Failures of either bricks or the joints lead to relining or reconstruction of the refractory wall. It has been well established that the weakest part of a refractory lining is the joints made up by mortars as per failure is concern. Thus in order to increase the performance of a refractory lining, scientists, and refractory professionals around the globe are engaged to develop high performance refractory mortar. The development of value added mortars relies on the scientific understanding of their performance evolved since their inception.

Mortar used to bind the bricks is low cost materials as compared to bricks. But its importance is much more than brick. Because it forms the lining between the refractory articles; if failure occurs, it causes the damage of the whole refractory system.Aggregates used in a mortar should be similar to the composition of brick to which mortar has to join but their particle size must not be similar as in the brick. Particle size of the aggregates must be less than 200 meshes. After the aggregate content the major content of mortar is the binder. Performance of mortar strongly depends upon the nature of the binder used in the formulation. So a suitable binding agent must be selected which provides green as well as fired strength to a mortar.

Bonding system of mortar highly depends upon its binder composition and the water content to give a required consistency. Normally, aggregates and binder has been mixed in a powder form and supplied to the customer and customers have to add water on it toachieve a required consistency so that it can be easily applied in between the bricks to join. Due to this water addition by the customer, water percentage varies customer to customer. With same aggregate content, these varying water percentages also vary mortars properties like strength; flowability etc. so to get over from this problem a ready mixed mortar has been invented by some professional scientists to give mortar a constant consistency and properties or easy application.

Binder type and its amount greatly influence the properties of mortar. Different type of binders are there and their uses are different and it affects while mix with aggregates in a different way. It also gives different properties while combined with water. So binder selection has much more importance while preparing a mortar. Binder selection has been

done by considering the types of binder, its properties, mainly its bonding characteristics. This binding characteristic of binder divides the mortar into 3 parts they are: air-setting mortar, heat-setting mortar, Hydraulic-setting mortar. In air-setting mortar, binder has been selected to give bond strength upondrying.Itmaintains the strength of the bond up to the temperature at which ceramic bonding takes place. In heat setting mortar, binder has been selected, so that it forms bond between aggregates at higher temperature sintering. At last, in hydraulic setting mortar binder must be chosen which chemically combine with the aggregates and forms a bond between them by forming chemical compounds.

This thesis aims to develop a 45% alumina containing alumino-silicate mortar with suitable binders and their properties has been studied with respect to the percentage of binders and to the temperature. Bentonite, Plastic clay and sodium silicate have been studied as a binder system in this mortar. Optimization of different binder content has been studied based on bonding strength of a brick joint. Physical and mechanical properties of the mortar thus developed have also been studied in detail.

The thesis has been divided in to 6 parts where chapter 1 describes a general introduction of the subject. Chapter 2 provides a detail analysis of the literatures available with the objective of the present work. Chapter 3 describes the detail methodology and experimental techniques used in the present study. Chapter 4 discussed the results of the present study. Finally the conclusion from the present study has been summarised in chapter 5.

Chapter 2

Literature review

2.1 Refractory Mortars

Refractory Mortar is a finely grounded refractory mixture used to bind refractory bricks usually after the addition of water and binder. It is used to fill the Refractory linings and seams. Refractory mortars consists mainly four components: aggregates, plasticizer, binder and water. Their composition usually must be compatible with the properties of the refractory bricks. A distinction is made among ordinary refractory mortars, which set at high temperatures as a result of the formation of a ceramic bond; hydraulically hardening mortars, which contain hydraulic cement; and mortars with chemical bonding, which set at room temperature or upon heating. Mixtures to which a non-ceramic binder has been added are called refractory cements [1].

The term Mortar is used to indicate a paste prepared by adding requisite amount of water to a mixture of binding materials like cement or lime and fine aggregates like sand. They are used for laying and bonding shaped refractory products such as bricks. They are normally applied by trowelling [2]. The two components of mortar, namely, the binding material and fine aggregates are sometimes referred to as the matrix and adulterant respectively. The matrix binds the particles of the adulterant. Thus, the durability, quality and strength of mortar will mainly depend on the quality and quantity of the matrix. The combined effect of the two components of mortar is that the mass which is able to bind the bricks or stones firmly [1-2].

The degree of fineness of a refractory mortar is determined mainly by measuring the aggregate grain size of the mortar. The grain size of fine-grained mortars does not go exceed 1 mm; that of the coarse-grained mortars is up to 2 mm. A solution of the required consistency, consisting of mortar and water, is usually prepared at the work site. [1]

Four characteristics are essentially required for a refractory mortar. They are good workability and reasonable hardening time, retain necessary refractoriness, resist separation from the bricks and cracking during drying and heating, maintain good joint strength under operating conditions so the brick work structure remains sealed [3-4].

2.2 Classification of refractory mortars:

Heat-Setting Mortar: Heat setting mortar set at high temperature by sintering or development of ceramic bond. This mortar is mix with Water to get the desired consistency without binder.

High strength develops upon firing at high temperature according to its chemical composition and refractoriness [3-5].

Air-Setting Mortar: This type of mortar develops its strength upon drying. It consists of a Mixture of Aggregate & Binder, either in wet or dry condition.Chemical binders maintain the strength of the bond up to the temperature at which ceramic bonding takes place. It i advantage in which a strong bond over a wide range of temperature is desirable [3-5].

Hydraulic-setting mortar: These are the mortars containing hydraulic cement so strength develops upon hydration of the cement. Such mortars are used for special parts that are exposed in water at low temperature [3-5]. According to the nature of application, the mortars are classified in to two categories.

Brick laying mortar: These mortars are prepared for brickworks and walls. They are commonly used for joining refractory articles. Depending upon the working condition, type of construction and requirement the composition of mortars with respect to the kind of binding material is selected [3].

Finishing mortar: These mortars include common plastering work. For finishing purpose the mortars are composed of suitable materials with consideration of mobility, water retention and resistance to atmospheric action [3]. Each of the above classes of mortar is further classified by the condition in which they are supplied:

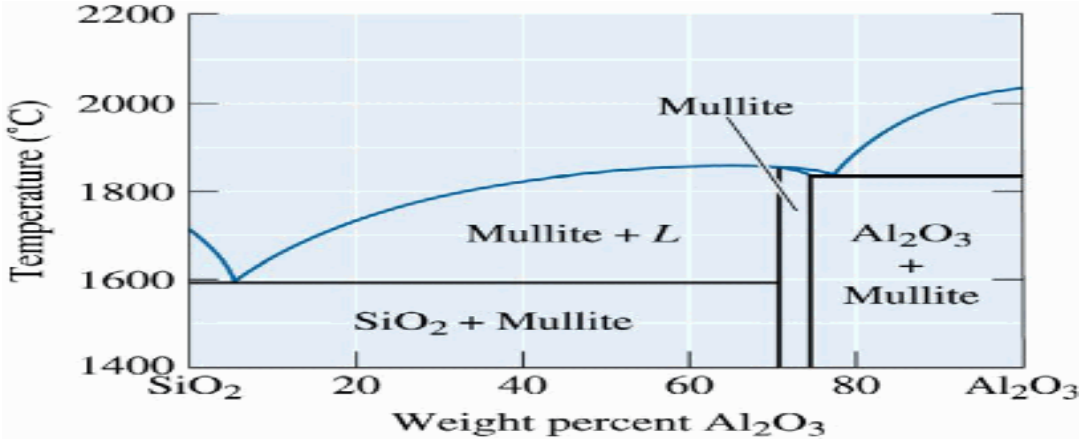
Dry Mortars: This class is supplied dry and must be mixed with water prior to use.Wet Mortars: This class is supplied in a ready for use [3].

Refractory mortars are used to bind the refractory units such as bricks, stones, to form an soft and even bedding layer for refractory units, to form joints, to improve the general appearance of structure, to serve as a matrix to hold coarse aggregates, to hide open joints of brick work, to fill up cracks detected in the structure during maintenance process and for linings of industrial furnaces and other equipment for example, firebrick and high-alumina mortars in blast furnaces, air heaters, and steel-casting ladles; dinas mortars in coke ovens; and magnesia mortars in open-hearth furnaces[3].

2.3 Raw materials and their selection for body formulation of refractory mortar:

In selecting each raw material, the consideration should be composition, uniformity, physical characteristics, Quantity, Location, Transportation, unit cost. Composition depends upon the types of mortar to be produced and the properties required. Main physical characteristics required while choosing refractory mortar that they should maintain uniformity between them and also posses uniform hardness across the product [6]. Second consideration is the binder added into composition and their properties.

2.3.1 Alumino silicate: Studies on Al_2O_2-SiO_2 phase diagram (Fig. 2.1)showed mullite ($3Al_2O_3.2SiO_2$) as the only stable compound of this system and reported that it melted congruently at 1828°C [7][8].



[Fig. 2.1 Al_2O_3- SiO_2 Phase diagram] [45]

It has been well studied that mullitization in mixture consisting of alumina and silica is influenced by the structure of the components used. The temperature at which these materials combine to give mullite are thus an indirect consequences of the structural stability. The formation of mullite starts from 1200^oC [9].

2.4 Alumino-Silicate Mortar

Refractory mortars mainly consist of fillers or aggregates, plasticizer and binder. Their composition usually must be compatible with the properties of the refractory bricks.

2.4.1 Aggregates: Fine aggregate (sand) is the main constituent of mortars. Some screeds incorporate a coarse aggregate (normally 10 mm maximum size) but generally their major

constituent is fine aggregate. Confusion sometimes exists over the difference between the terms fine aggregate and sand, there is in fact no difference the two words may be interchanged. Fine aggregate quality is affected by a number of factors: The mean particle size, grading, presence of impurities, shape and texture of the particles. The selection of suitable aggregates, which are capable of producing a product with the optimum properties, is most important. The design of mortar mixes is based on the concept that the voids in the fine aggregate, which are generally in the range 25- 40%, will be filled with binder. Where the voids are not completely filled there is an increasing risk that the mortar will not be durable. Aggregate may be natural, manufactured or recycled [3] [4] [10].

The fire clay is refractory clay which is capable of resisting a high temperature without being melted or softened and without losing its shape. Thus the fire-clay is used in manufacture of fire bricks, crucibles, lining materials for furnaces etc. The constituent of a good fire clay are two- alumina and silica. Percentage of alumina varies from 25 to 30 and silica from 75 to 65 [3-4].

Bauxite is an ore consisting of boehmite (AlO (OH)) a monohydrate of alumina and gibbsite ($Al_2O_3.3H_2O$), a tri-hydrate of alumina. Bauxite may contain these products in different proportion. Bauxite is a raw material rich in alumina and low in alkalis, alkaline earth and silica. Bauxite used as refractory raw material has high alumina content and low impurities. Diaspore($Al_2O_3.H_2O$) is also monohydrates of alumina formed by hydrothermal metamorphism of silicate minerals. It forms $\propto$-alumina directly when heated without forming an intermediate product. Raw material of alumina transported from china contained a large proportion of diaspore. So these bauxite are called Chinese bauxite [11] [12] [17].

Sillimanite group of minerals ($2Al_2O_3.3SiO_2$) are formed when aluminium silicate hydrates undergo thermal metamorphism at a high pressure. Sillimanite group of minerals decompose when heated into mullite and silica. But the three members of the group sillimanite, andalusite and kyanite decompose however at different temperatures. Sillimanite decomposes at 1530-1525°C. Mullite formed when these minerals decompose into a low density product. Sillimanite do not change greatly in volume when transformed to mullite [4].

2.4.2 Plasticizer: Plasticizers or dispersants are additives that increase the plasticity or fluidity of a material.Plasticizers or water reducers are chemical admixtures that can be added to mortar mixtures to improveworkability. Unless the mix is "starved" of water, the strength of mortar is

inversely proportional to the amount of water added. In order to produce stronger mortar, less water is added (without "starving" the mix), which makes the mortar mixture less workable and difficult to mix, necessitating the use of plasticizers, water reducers, super plasticizers or dispersants. The properties of other materials are also improved when blended with plasticizers including concrete, mortars, and related products.Plasticizers make it possible to achieve improved compound processing characteristics, while also providing flexibility in the end-use product. It evaporates at high temperatures and imperts a strong bond between the aggregates [13]. A plasticizer makes the mix flow better.it allows the mortar to be more fluid easier to spread, especially when brick laying, it slows the setting time down slightly, but it is used to allow the mortar to be just smoother.

2.4.3 Binders: Binders play an important role while preparing a mortar. It binds mortar aggregates due to which strength develops and provides the bond for the brick. Organic or inorganic binder, both powder and liquid are used. Organic binders are mainly used to improve the strength after drying and for adjustment of the bonding time. Sodium silicate is a typical liquid binder which is use mostly for wet type mortars. It has high bonding strength compared with a powdered binder and used in application where high strength is required after drying [3] [5].

Plastic clay is an extremely rare mineral found in very few places around the world. It is also sometimes referred to as 'ball' clay [3]. The main advantages of plastic clay are their high plasticity and dry strength [14]. Plastic clays usually contain three dominant minerals: from 20-80% kaolinite, 10-25% mica, and 6-65% quartz. In addition, there are other 'accessory' minerals and some carbonaceous material present as an impurity. The wide variation both in mineral composition and in the size of the clay particles results in different characteristics for individual clay seams within a deposit [15].

Bentonite is one of the clay containing montmorillonite as its Principal constituent. Montmorillonite ($Al_2O_3.4SiO_2.6H_2O$) is a three layer mineral like pyrophillite. Generally, part of Al^{3+} ions are replaced by Mg^{2+} ions and water molecules plus Ca^{2+} or Na^{2+} ions are absorbed between the layers. Thus chemical analysis shows that bentonite always contains a little CaO and Na_2O. If dried, the water molecules between the layers are removed. However Bentonite absorbs water again, water molecules are taken in between the layers, expanding them and thereby cause swelling. This swelling is less prominent in the bentonite absorbing a

large quantity of Ca^{2+}, but is high in bentonite with a large proportion of Na^{2+}. The former is referred to as calcium bentonite and latter as sodium bentonite [16-22].

Bentonite-water mixtures are widely used for bonding and plasticizing agents in ceramic fabrication. A bonding agent should have certain adhesive properties, and these aredeveloped when bentonite is mixed with small amount of water.For application as a bonding agent, generally both water and clay are mixed into the finished product in amounts less than5 percent by weight [18-22].

A clay-water mixture becomes highly plastic when water content is in excessof that which is bound as "oriented" water by the bentonite. The main requirement is for the developmentof a plastic state adequate to allow the raw materials to be easily content happed ormoulded into the form of the finished product. Frequently a material isneeded which will also act as a bonding agent or binder of other ingredients.Therefore, small bentonite additions, which constitute a small percentage ofthe entire product, function both as plasticizing and bonding agents. Appliedin this fashion, the beneficial properties of bentonite are utilized; however, the addition is sufficiently small so that there is no adverse effect on thefinished products [23-25].

Bentonite is a clays containing montmorillonite as its principal constituent. It gives high plasticity to the mortar due to the big surface of the adhering water films [4]. It commonly used in mortar to bind the aggregates as a bonding agent and create a closed pack structure at high temperature. Layer structure of clay expands after wetting. Na^{2+} and Ca^{2+} present in bentonite are strongly hydrates in presence of water resulting in a hydrophilic environment in the clay surface [18-22].

Bentonites disperse into colloidal particles and accordingly provide large surfaceareas. This large surface area is a major reason why bentonitefunctions so well in stabilizing emulsions or as a medium to carry other chemicals. Chemically, montmorillonite is described as a hydrous aluminum silicate containing small amounts of alkali and alkaline earth metals [18-25].

Figure 2.2 showed the layered structure of montmorillonite. Montmorillonite adsorbs water whenever it is available due to its layer structure. Water adsorption occurs to the greatest extent on the basal surfaces of the clay and in this fashion pries adjacent flakes apart, resulting in overall volume increase of the clay. Evidence of this swelling mechanism is seen as an increase in the vertical axis dimension of the clay. The water which is adsorbed primarily on the basal surfaces of the clay consists of a regular, rather than a random,

arrangement of water molecules. The viscosity of an aqueous medium is increased substantially when bentonite is added. The bentonite that binds these products well is one which disintegrates into extremely small particles, provides a large available surface area, and allows maximum contact with other product components. In this way pores of the suspension are removed due to which density of the suspension increases [23-25].

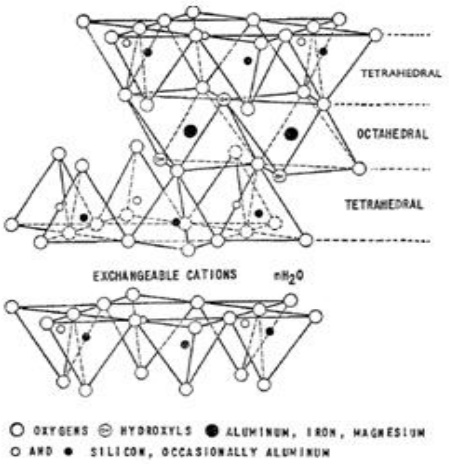


Fig. 2.2 Layered structure of Montmorillonite [23]

The Bentonite is a very fine grained material largely composed of the clay mineral, montmorillonite. While mix with some other substance it undergo isomorphous substitution. Montmorillonite lattice is negative in charge, owing primarily to isomorphous replacements of ions within the structure. This negative charge is balanced by cations which are held on the surface of the flakes. Cations held in this fashion by theclay can be readily exchanged; the cations most commonly found in natureare sodium and calcium.Bentonite particles in a suspensionas a bonding agent are disaggregated into thecolloidal particle range. Therefore, they have similar properties, and respondlike other colloidal suspensions [19-25].

Chemically, montmorillonite is described as a hydrous aluminium silicate containing small amounts of alkali and alkaline earth metals. Structurally, montmorillonite is made of two basic building blocks, the aluminum octahedralsheet and the silica tetrahedral sheet. A single montmorillonite unit cell consists of two silica tetrahedral sheets, between which is an aluminum octahedral sheet. Due to this concept bentonite gives a doble-layer structure and absorbs water into the layer. When a relatively small amount of water is mixed with a large amount ofbentonite, the mixture forms a sticky mass which has adhesive properties.This

physical state results when the water content is inadequate to fulfil theoriented water requirement by the montmorillonite. The plastic propertiesof a clay-water system increase as the ratio of water to clay increases. Each montmorillonite flake has adsorbed its maximum amount of water and additional amount of water in the system acts as a lubricant for the hydrated particles. After water addition, Bentonite disaggregates in water and swelling takes place, theaverage clay particle size decreases. As a function of decreasing particle size,the number of particles per unit weight of clay increases, and consequentlyalso the available surface area. It gives higher packing of aggregate particles and reduces pores in between them due to which strength increases. It could be seen from above picture that with increase in bentonite increases the strength of mortar [18-25].

A refractory mortar consists essentially a refractory aggregates, such as, ceramic grog, a bond clay and some water to produce enough plasticity for towelling or other methods of handling. Upon drying the ceramic aggregates are bonded by the clay in the mixture. At temperatures below actual vitrification of the mass, the clay is rather weak in nature and, therefore, the clay mortar has certain service limits which may in some cases significantly reduce its usefulness. In order to prevent the development of a bond having low strength at temperatures below red heat, certain additional binders, such as organic or inorganic bonding substances may be added to the mortar to achieve a cementing of the clay and grog into a more strongly bonded mass. Sodium silicate has been added to achieve such cementing action. It is well known that the sodium silicate may be added to such mortars either in form of a solution or in dries water soluble form, thus producing either a wet or a dry mortar [26] [27].

Sodium silicate is the common name for a compound sodium meta-silicate, Na_2SiO_3, also known as water glass or liquid glass. It is available in aqueous solution and in solid form and is used in cements, passive fire protection, refractories, textile and lumber processing, and automobiles. Sodium carbonate and silicon dioxide react when molten to form sodium silicate and carbon dioxide [28] [33-36]:

$$Na_2CO_3 + SiO_2 \rightarrow Na_2SiO_3 + CO_2$$

Anhydrous sodium silicate contains a chain polymeric anion composed of corner shared $\{SiO_4\}$ tetrahedral, and not a discrete SiO_3^{2-} ion. In addition to the anhydrous form, there are hydrates with the formula $Na_2SiO_3 \cdot nH_2O$ (where n = 5, 6, 8, 9) which contain the discrete, approximately tetrahedral anion $SiO_2(OH)_2^{2-}$ with water of hydration. For example, the

commercially available sodium silicate pentahydrate $Na_2SiO_3 \cdot 5H_2O$ is formulated as $Na_2SiO_2(OH)_2 \cdot 4H_2O$ and the nano-hydrate $Na_2SiO_3 \cdot 9H_2O$ is formulated as $Na_2SiO_2(OH)_2 \cdot 8H_2O$ [28] [29] [30].

In industry, the different grades of sodium silicate are characterized by their SiO_2:Na_2O ratio, which can vary between 2:1 and 3.75:1. Grades with this ratio below 2.85:1 are termed 'alkaline'. Those with a higher SiO_2:Na_2O ratio is described as 'neutral' [28-33].

History of Water Glass was defined in Von Wagner's Manual of Chemical Technology (1892 translation) as any of the soluble alkaline silicates, first observed by Van Helmont in 1640 as a fluid substance made by melting sand with excess alkali. Glauber made what he termed "fluid silica" in 1648 from potash and silica. Von Fuchs, in 1825, obtained what is now known as water glass by treating silicic acid with an alkali, the result being soluble in water, "but not affected by atmospheric changes". Von Wagner distinguished soda, potash, double(soda and potash), and fixin as types of water glass. The fixing type was "a mixture of silica well saturated with potash water glass and a sodium silicate" used to stabilize inorganic water color pigments on cement work for outdoor signs and murals [28-36].

Properties of sodium silicate are a white powder that is readily soluble in water, producing an alkaline solution. It is one of a number of related compounds which include sodium orthosilicate, Na_4SiO_4, sodium pyrosilicate, $Na_6Si_2O_7$, and others. All are glassy, colourless and dissolve in water.Sodium silicate is stable in neutral and alkaline solutions. In acidic solutions, the silicate ion reacts with hydrogen ions to form silicic acid, which when heated and roasted forms silica gel, a hard, glassy substance [32] [36].

Refractory use Water glass is a useful binder of solids, such as vermiculite and perlite. When blended with the aforementioned lightweight aggregates, water glass can be used to make hard, high-temperature insulation boards used for refractories, passive fire protection and high temperature insulations, such as moulded pipe insulation applications. When mixed with finely divided mineral powders, such as vermiculite dust (which is common scrap from the exfoliation process), one can produce high temperature adhesives. The intumescence disappears in the presence of finely divided mineral dust, whereby the waterglass becomes a mere matrix. Waterglass is inexpensive and abundantly available, which makes its use popular in many refractory applications [28-36].

The water glass favours the polymerisation process, leading to a reaction product with more Si and more mechanical strength. As water is removed from liquid silicate, the silicate progressively becomes tackier and more viscous. The removal of a relatively small amount of water will render the liquid silicate a glassy film while air drying [34] [35]. When fired at higher temperature it forms glassier phase which binds aggregate material together to form a highly compact structure which increases its strength more and more. Sodium silicate as binder gives equal strength amount at air drying which clay binder gives upon firing at lower temperature. Above graph shows the increase in temperature with increase in sodium silicate content [28-35].

Alumino-silicate Mortars:

J. Goswami et. al. [39] prepared a mortar by using sodium silicate as a binder which explains that bonding strength of a mortar containing sodium silicate as a binder increases as a function of binder content as well as with increase in temperature. But strength of mortar increases only to a certain extent of firing temperature after that it decreases due to high amount of liquid phase formation.

An investigation about a composite binder containing liquid glass as its main component describes that the low-density liquid glass produces a strengthening effect on the films that coat refractory components due to which moisture cannot pass through it. Because of this moisture content, strength of the product decreases. By using alumina cement the hardened thin hydrate films created which prevent the swelling of the composite binder on heating at low temperature [37].

R. Stonis₁et al. [38] investigated about a composite binder composed of water glass with hardener and high-alumina cement which is used to prepare an air-setting refractory mortar based on fireclay fillers. Components of a composite binding mortar used are water glass (a sodium silicate solution) with a hardener, high-alumina cement, and chamotte filler. A hardener used for water glass was an iron-chromium slag (ICS) containing about 85% dicalcium silicate C2S (γ-CaO. SiO_2). It was found that with decrease in ICS percentage, consumption of water glass decreases and setting-up time increases due to whichsmaller shrinkage strain was observed. It was also found that increase in amount of liquid sodium silicate and hardener will increase the shrinkage of the sample. The larger the amount of

hardener (dicalcium silicate) and consequently of water glass the larger the shrinkage strain. Refractory mortar with a composite binder using water glass of density 1.25 g/cm^3 was found to be best among other composition due to its superior strain characteristics and its high temperature application [38].

N.I Filimonova et. al. [40] investigated the effect of phosphate binder on mullite-zirconia-corundum composition to join mullite-corundum bricks. Aluminaborophosphate is used as a phosphate binder of density 1.56 gm/cm^3 in the literature. This aluminaborophosphate binder gave high shear strength to the mortar at higher firing temperature due to which it's binding property increases as a function of firing temperature [40].

L. A. Babkina, 1 M. I. Prokopenko, 1 L. N. Soloshenko [41] investigated about the use of phosphate binder in a corundum mortar. Short supply of orthophosphoric acid limits its use as a phosphate binder. Thus binders like LST, technical H_3PO_4, technical sodium polyphosphate, and technical sodiumtripolyphosphate has been used and their properties have been tested. Maximum strength of cohesion at high refractoriness has been given by the composition contains an aqueous solution of H_3PO_4 with a density of 1.40 g/cm^3 and LST as a binder. Strength of cohesion reduces with the reduction of the density of the aqueous solution of sodium try poly phosphate (Na (PO_3)). $Na_5P_3O_{10}$ cannot be used as a binder in the composition of corundum mortar due to its low cohesion strength. High cohesion strength is the main goal of the present work. These binders were salts of orthophosphoric acid, i.e., Na(PO_3) and $Na_5P_3O_{10}$. Aqueous solutions of H_3PO_4 cannot be used because of its intense interaction with periclase. Their study has been shown that the introduction of phosphate binders into the mortar intensifies the process of its sintering with the lining without deteriorating the high refractoriness. Use of higher content of the additive increases strength insignificantly [41].

Keun-Hyeok Yang and Jin-Kyu Song [38] investigated about different alkali activated by-products like fly ash, ground granulated blast furnace slag and condensed silica fumes used as binder materials in mortars. A sodium hydroxide solution or sodium silicate solution is used as a good activator. A paste activated by alkali solutions experiences very short setting times and subsequent shrinkage. This would cause rapid workability loss of the mortar with time, which would constitute an obstacle for practical applications. They studied the use sodium silicate powder and sodium hydroxide powder as the activator. The compressive strength of the ground granulated blast furnace slag (GGBS) based alkali activated mortar was greatly

influenced by the ratio of sodium oxide (Na_2O) of the activator to the source material (SM) by weight, and the type and fineness of the ground granulated blast furnace slag (GGBS). Sodium hydroxide powder was added to control the molar ratio (SiO_2/Na_2O) of the sodium silicate. The mechanical strength of the alkali activated (AA) pastes depends on the silicon oxide-to-sodium oxide ratio (SiO_2/Na_2O) in the alkaline activator, and the silicon oxide-to-aluminium oxide (SiO_2/Al_2O_3) ratio and calcium content in the source material. The initial flow of the alkali activated (AA) mortar decreased slightly with theincrease of the amount of sodium silicate but was little influencedby the amount of sodium hydroxide.

OBJECTIVE:

A detail literature study shown that binder plays an important role in the development of good quality refractory mortar. Properties of refractory mortar are strongly dependent on the type, nature and amount of binder added. In view of this present study, it aims to develop 45% Al2O3 mortar with suitable binders. These binders are bentonite, plastic clay and sodium silicate liquid. The specific objectives of the present study are as follows:

1. Study the effect of bentonite content on the properties of a 45% Al2O3 mortar
2. Study the effect of plastic clay content on the properties of a 45% Al2O3 mortar
3. Study the effect of sodium silicate liquid on the properties of a 45% Al2O3 mortar

Chapter 3

Experimental and procedure

3.1 Raw materials used in the present study:

Alumino-silicate(Al_2O_3-SiO_2) mortar having 45% Al_2O_3has been prepared by takingmain constituent of alumina silicate like Chinese bauxite, fire clay grog, silliminite sand as base material or aggregates andclay minerals like Plastic clay and bentonite as plasticizer or binder. Attempt has also been made to prepare mortar formulation with sodium silicateas a binder. Typical Al_2O_3 content of the aggregates and binders used are tabulated in Table III.I

Table III.I Al_2O_3 Content of Aggregates and Binders

Raw Materials	**Alumina Content (%)**
Chinese bauxite	80
Fire clay grog	38
Silliminite Sand	58
Plastic Clay	26
Bentonite	15

Typical characteristics of Sodium silicate Liquid is as follows:

Table III.II Typical characteristics of Sodium silicate Liquid

Sodium Silicate liquid Binder	
SiO_2	20%
Na_2O	6%
Na_2O/SiO_2 Ratio	1:3
Sp Gravity	1.25 gm /cm^2
Solid Content	30%

3.2 Mixing of aggregates and binder and making of mortar paste

Mixing of raw materials is an important step to prepare a mortar with better homogeneity, consistency, which influences the properties of mortar. The required amount aggregates has been weighted properly along with binder and plasticizer. After dry mixing required amount of water was added and then mixed for some time in order to prepare a paste like structure having good consistency so that it can be easily applied on brick structures. The finer fractions of the batch composition were first dry mixed separately in the Hobart mixer (Fig. 3.1) also it can be hand mixed by using a trowelin order to prepare a homogeneous powder.

This apparatus consist of pot and agitator. The pot always remains in static position and the agitator rotates around the pot and also about its own axis.

[Fig. 3.1 Hobert Mixture used in the mixing the aggregates and binder with water] [43] [44]

3.3 Flowability of mortar

Flow behaviour of mortar is one of the most important properties related to the installation technique of the mortar. It depends fully on the particle size distribution, shape and size of the aggregate and binders used in the body formulation of the mortar. Flow behaviour of the mortar has to be measure to determine the performance of the mortar as well as the required percentage of water to achieve 100% flow. Flow value of mortar measured using ASTM standard flow table test (ASTM C230) [4]. Flow cone is completely filled with the mortar prepared so that no air bubble presents inside. There after the brass cone is removed and mortar paste is allowed to spread by the action of vibration which is given by tapping. Percentage increase in diameter of spread is measured after the elapse of one minute. Flow value was expressed as percentage increase of the diameter of mortar. The conical brass cone had inner diameter 100mm and 70mm respectively at a height of 50 mm. Typical brass cones and tap flow measuring instrument has been shown in Fig. 3.2.

Flow value has been calculated using Eq. (3.1):

$$\text{Flow percentage of mortar } (\%) = \text{ Average material flow diameter} - 100$$

(a) (b) (c)

Fig. 3.2 a) Mortar in conical frustrum b) prior to dropping table c) after 15 taps

3.4 Joint Stability of mortar

Joint stability is the ability of mortar to bind the refractory bricks. After preparation of mortar it is applied on bricks to check the joint stability [45]. It is the ability of mortar to be easily placed and spread or it is a measure of the ease with which a fresh mix of mortar can be handled and placed [43]. Or, The ease with which a given set of material can be mixed in to mortar and subsequently handled, transported, placed and finished with a minimum loss of homogeneity [44]. For this test 3 refractory standard alumina brick was taken and after preparation of mortar it was applied on every brick in appropriate amount so that it joints with other brick properly.After jointing the bricks they were kept for 15 minutes after that there jointing stability was checked.

[Fig.3.3 Photographs showing the joint stability]

3.5 Casting of mortar

The test samples were casted in metallic mould required size in order to test physical andmechanical properties. Mouldshave been lubricated by greasebefore casting. Mortar has been casted in160×40×40mm iron mouldfor the measurement of Cold Modulus of Rapture and linear shrinkage, Cold crushing strength, Apparent Porosity and BulkDensity measurements. As a mould lubricant, paraffin oil has been used. For different testing three samples were casted for each batch.Vibrating table was used for vibration to remove air bubbles and excess materials to give a perfect shape to the mortar having no air in between the material which decreases its strength by creating more no of voids while drying.

3.6 Drying and Firing of samples

Samples were allowed to air dry undisturbed at room temperature for 24 hours and then oven dried at 110˚C for atleast 12 hours.Dried samples were fired at 1000°C,1300°C and 1500°C with constant firing with soaking time around 3 hours in an electric arc furnace. Above 1000°C, densification ofmortarstarted due to the formation of ceramic bond that cause of high strength of themortar and amount of water added has been removed completely gives a closely packed structure.

3.7 Characterization of samples

3.7.1 Drying and firing shrinkage

Ceramic samples undergo drying and firing shrinkage during drying and firing processes. This expansion and shrinkage of the sample is occurring due to either phase transformation or densification and it is expressed in %. Test samples for this measurement was 160×40×40 mm. Samples were dried at 110°C and fired at 1000°C,1300°C and 1500°C with 3 hours soaking time. In this measurement, initial length was taken before drying the sample. After that, the samples were fired in the furnace and final length was measured. Percentage linear change (PLC) was calculated by given formula.

$$\text{PLC(in \%)} = \frac{\text{Initial Length} - \text{Final Length}}{\text{Initial Length}} \times 100$$

3.7.2 Apparent porosity:

Apparent porosity of the sample defined as the ratio of open pores to the bulk volume of the material and expressed in percentage. Measurement of bulk density was 60×40×40 mm and tested by IS 1258 vacuum method. First dry weight of sample taken after that put in to air tight desiccators for 30 minutes in vacuum condition. After 20 minutes, water was introduced in the desiccators for 15 minutes. The samples were fully enclosed with water and to fill up the pores. After that, socked and suspended weight was taken. Apparent porosity of the samples has been calculated using the formula give below.

$$\text{Apparent Porosity} = \frac{\text{Soaked Weight} - \text{Dry Weight}}{\text{Soaked Weight} - \text{Suspended Weight}} \times 100$$

3.7.3 Bulk Density:

Bulk density of thesample is inversely related to the porosity of the same sample. The bulk density is generally considered in conjunction with apparent porosity. It is a measure of the weight of a given volume of the refractory. Bulk density of the sample defined as the ratio of mass of dry material to the bulk volume of the sample and expressed in gm/cc.

$$\text{Bulk Density} = \frac{\text{Dry Weight}}{\text{Soaked Weight} - \text{Suspended weight}}$$

3.7.4 Cold crushing strength

Cold crushing strength (CCS) is defined as the maximum amount of compressive stress per unit area that causes fracture in the refractory material. It measures the strength of mortar as well as strength of its bonding system as expressed in Kg/cm^2. Semi automatic hydraulic compressive testing machine apparatus used for this measurement. Load was applied on the flat surface of the sample and measure the load at which crack was propagating in the refractory sample. Cold crushing strength calculated by given formula.

$$\text{Cold Crushing Strength in Kg/cm2} = \frac{\text{load}}{\text{area}}$$

3.7.5 Cold modulus of rapture and bonding strength:

The cold modulus of rupture of a refractory material designates the bending strength and its suitability for use in construction of furnace lining and as expressed in $Kg/cm2$. This Modulus of rapture measurement was determined by three point bending test (ASTM C233) with the

sample size was 160×40 mm and the span length should be 100mm. During testing, load should be uniform and applied at centre of the spam length. Using given formula cold modulus of rapture was calculated.

$$\text{Cold modulus of rupture in Kg/cm}^2 = \frac{3wl}{2bd^2}$$

Where,

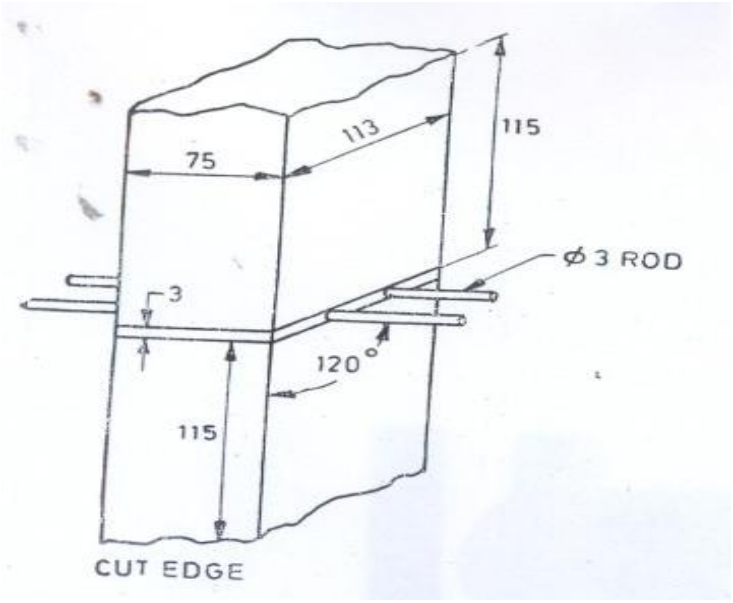
W= facture load (in Kg)

l= span length

b= width of the sample

d= thickness of the sample at fracture plane

Bonding strength of the joints formed has been measured using IS 11452. The different steps of sample preparation have been shown in Fig. 3.4. Four standard bricks are selected of size 250×113×75 mm having modulus of rupture higher than that anticipated in the mortar. Each of these four bricks was cut in to two equal parts on a plane parallel to the 75×113 mm face. The uncut 75×113 mm face of each brick is used to form the mortar joint. Mortar of different composition are applied on the uncut face are squeezed to form to 5-6 mm thickness and is squeezed to form a joint of 2 mm in thickness of the uncut and horizontally placed face of one half brick. This is facilitated by placing two spacer rods in the mortar parallel to the 75mm edges of the brick. Uncut face of other half brick is placed on the mortar. The excess mortar in the joint are forced out by pressing on the top half of the brick and at the same time moving it with a to and fro motion in the direction of 113 mm dimension of the brick using the spacer rod as rollers. The rods are withdrawn and the excess mortars were cleaned from the surface of the joint. The strength of the joint has been measured using three point bending method using the formula discussed earlier. Typical arrangement for sample for this measurement is shown in Fig. 3.5.

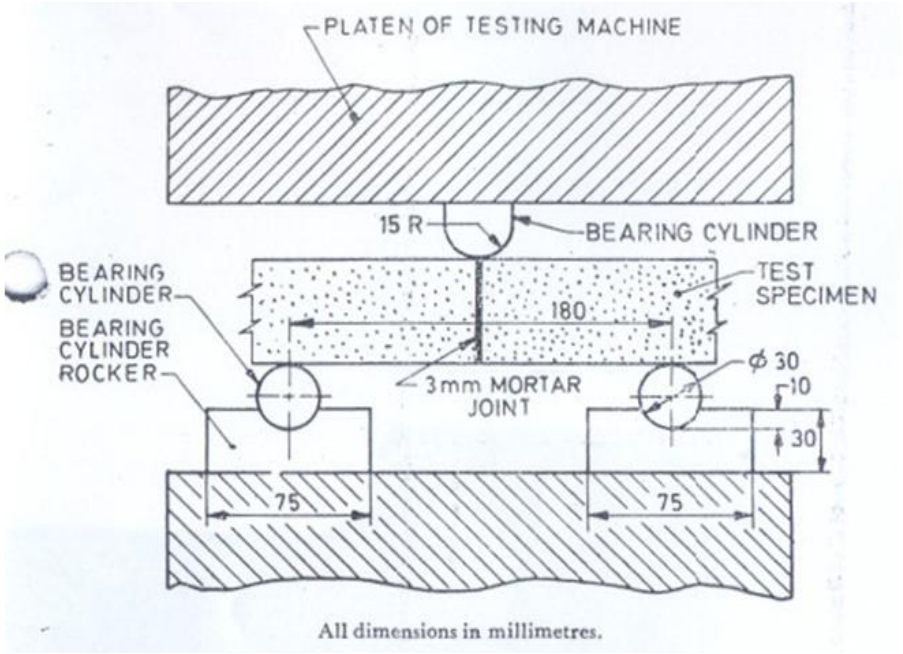


Method: IS11452

STEP 1 STEP 2

STEP 3 STEP 4

[Fig. 3.4 Steps for sample preparation to measure joint strength] [39]



[Fig. 3.5 Sample arrangement for joint strength measurement] [IS : 11452 -1985]

3.7.6 XRD Phase analysis:

Phase analysis is carried out by powder diffraction method by Rigaku, Miniflex II Japan XRD instrument. Mortar samples fired at different temperature were broken into small pieces and ground in to fine powder. Different phases present in the fired powder sample were determined by XRD pattern. X-ray generator was set at 35kV voltage and 15 mA current. Cu K_α radiation with $\lambda = 1.54$Å has been used as incident radiation. The samples were scanned between 10-80^o at a scan speed 0.02^o per second. Analysis of obtained diffraction pattern was done by matching them with different XRD patterns by using Philips X-pert high score software.

Chapter 4

Results and Discussion

4.1 Batch formulation of alumina silicate mortar having 45% Al_2O_3 with different Bentonite content:

4.1.1 Batch formulation of 45% Al_2O_3 mortar with bentonite as binder

Raw materials used in the mortar formulations are as follows. Aggregates: 80% Chinese Bauxite, Fire clay Grog, Silliminite Sand. Binders: Plastic clay and Bentonite. The effect of bentonite on the properties of mortar has been studied on the basis of linear shrinkage, strength and other properties. In the present study, four different formulations have been prepared by varying bentonite. Batches formulation of alumina-silica mortar has been shown in Table IV.I with different bentonite percentage.

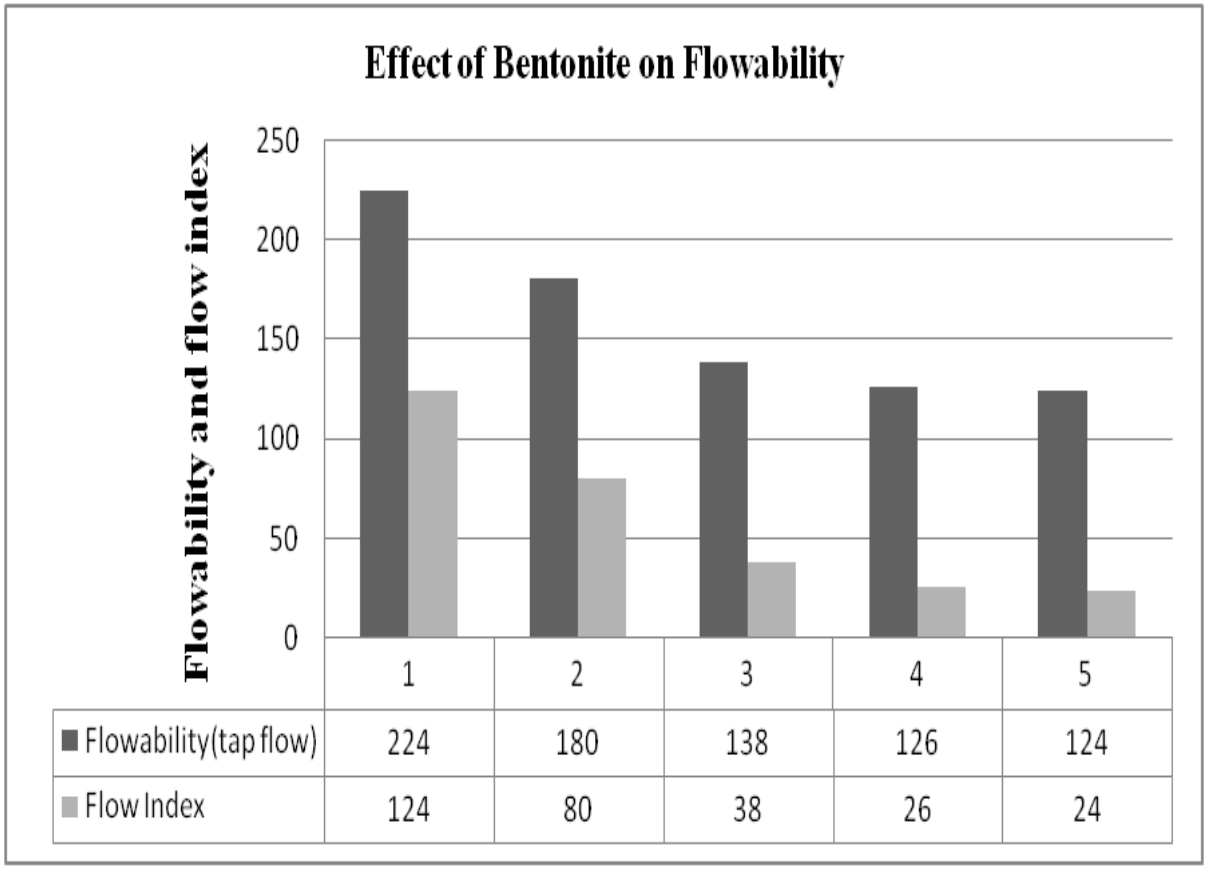
Table –IV.I Batch formulation of 45% alumina mortar with Bentonite as binder

Composition	T2	T-5	T-6	T-7	T-8
Fire clay grog (-200 mesh)	35	36	37	38	39
Silliminite sand (-200 mesh)	20	20	20	20	20
Chinese Bauxite (-200 mesh)	20	20	20	20	20
Bentonite	5	4	3	2	1
Plastic clay	20	20	20	20	20

4.1.2 Flowability of 45% Al_2O_3 mortar prepared with bentonite binder

Variation in flow at constant water percentage has been studied as a function of bentonite percentage graphically presented in Fig. 4.1. Bentonite is a clay containing montmorillonite as its principal constituent. It has a layer structure. The layer structure expands on wetting. Sodium and calcium ion present in bentonite can easily exchange with other ions in slurry leading to the formation of hydrophilic environment on the clay surface. Water added to incorporate flowability of the mortar is utilized in two ways. One part of the water fill up the

interstitial voids between the aggregates and the other part forms a water film on the surface of the aggregate. The more the second part water the more will be the flowability of the mortar. In the present study all the compositions have same constituents except a small variation in bentonite content. Interstitial porosity of the aggregate is constant for all the composition so water required for filling up the interstitial pores is constant for all the cases. Bentonite adsorbs some amount of water for its expansion. More the amount of bentonite is more amount of water is adsorped. Water remains to form the water film on the aggregate decreases with increase in bentonite content. Thus the sample prepared with more amount of bentonite (5%) shows less flowability as compared to that prepared with low amount of bentonite.

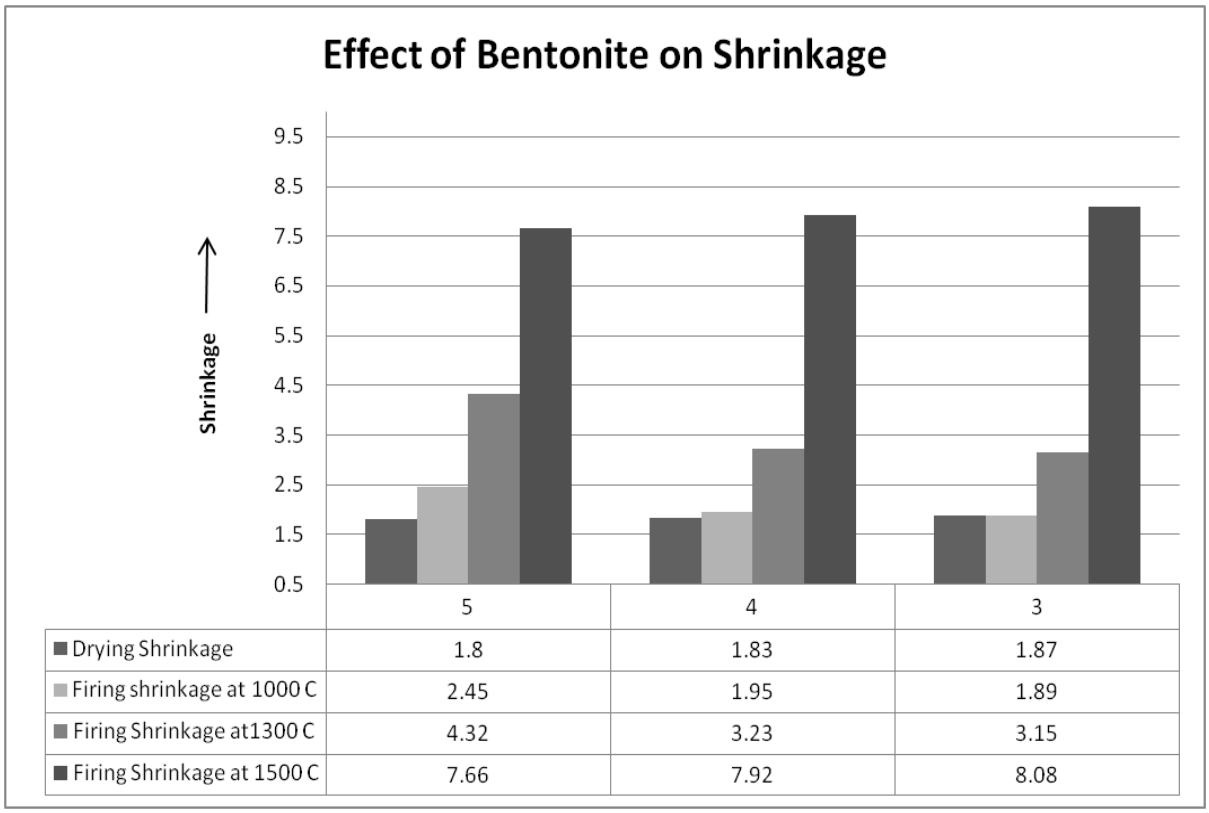


[Fig. 4.1 Flow behaviour of 45% alumina mortar as a function of bentonite]

4.1.3 Shrinkage Behaviour of 45% alumina mortar prepared with bentonite binder

Shrinkage behaviour of mortar as a function of bentonite at 110^{o}C, 1000°C, 1300°C and 1500°C has been shown in Fig. 4.2. It could be seen from the above graph that drying

shrinkage of the sample decreases with increase in bentonite clay content. It has been discussed earlier that when bentonite added to the composition it disintegrates in to small particles to fill up the pore between the aggregates. When water has been added in to the formulation due to hydration properties bentonite absorbs some amount of water due to its dual-layer structure. This water adsorption expands the bentonite which fills up the voids present in the sample. Drying shrinkage comes out from the removal of water film from the aggregate and the shrinkage due to drying of expanded bentonite. The removal of moisture from the interstitial void does not provide any drying shrinkage.



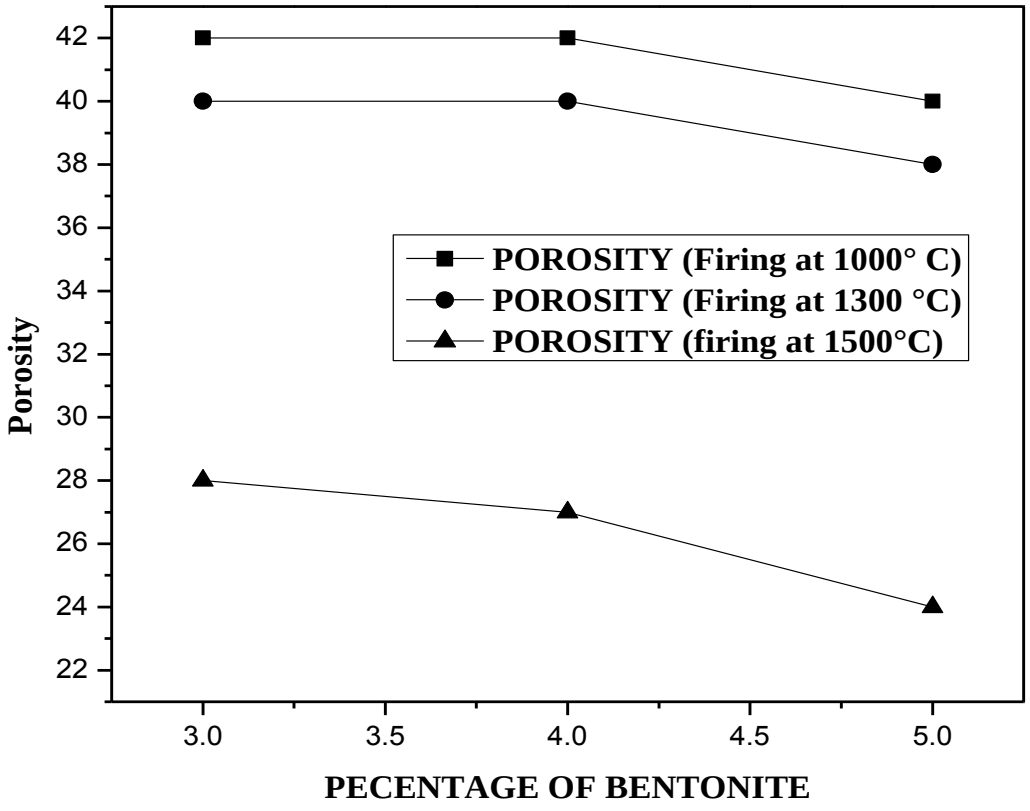
[Fig. 4.2.Shrinkage behaviour of 45% Al_2O_3mortar as a function of bentonite and temperature]

The contribution of drying shrinkage arising from the removal of the water film is more as compared to that due to shrinkage of expanded bentonite. So, low bentonite content gives more shrinkage as compared to higher bentonite. At 1000^0C the firing shrinkage shows a very small increment for all the samples. This increment is little bit higher for the sample prepared with 5% bentonite. Bentonite is a low melting compound which gives rise to more shrinkage in the sample. Shrinkage rate increases with increase in temperature. This increase in shrinkage is attributed to the densification of the sample. Shrinkage rate increases with

increase in temperature for all the samples. This is attributed to the liquid phase sintering of the sample.

4.1.4 Apparent porosity of 45% alumina mortar prepared with bentonite binder

Apparent porosity of the mortar as a function of temperature and amount of bentonite has been shown in Fig.4.3. It could be seen from the above graph that the apparent porosity varies very less as a function of bentonite content as well as at different temperature. It could also be seen that with increase in temperature, porosity decreases. This is attributed to the densification of the sample as a function of increase in temperature, which is quite obvious. Drying shrinkage as well as firing shrinkage of the mortar prepared with high bentonite content was more as compared to low percentage of bentonite.



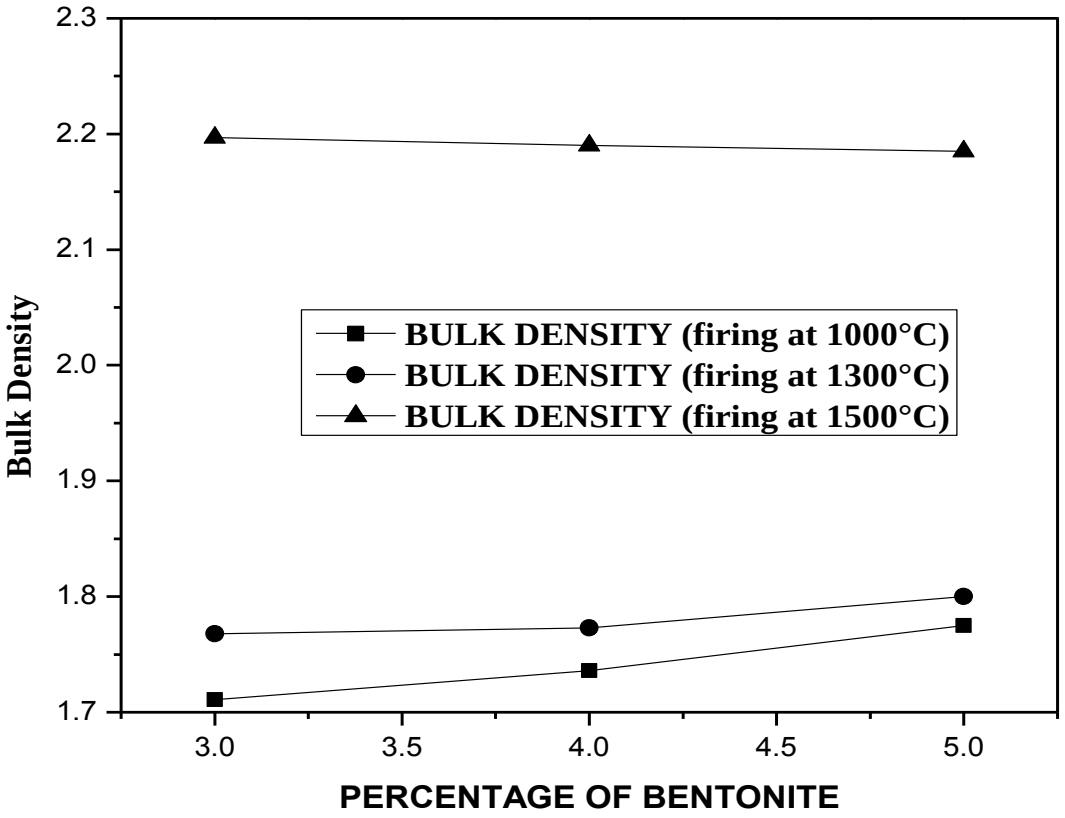
[Fig.4.3. Apparent porosity of 45% Al_2O_3mortar as a function of bentonite and temperature]

As a result apparent porosity of the sample decreases as a function of bentonite content. Rate of increase in porosity as a function of increase in temperature is more. This is attributed to

the enhanced liquid formation at high temperature which contributes enhance densification in the sample leading to more decrease in porosity.

4.1.5 Bulk density of 45% alumina mortar prepared with bentonite binder

Bulk density of the mortar as a function of temperature and bentonite content has been shown in Fig. 4.4.



[Fig. 4.4 Bulk density of 45% alumina mortar as a function of bentonite and sintering temperature]

It could be seen from the figure that bulk density follows an inverse strain as that of apparent porosity. As all the samples have been prepared with same aggregates except a small variation in bentonite content they are likely to have similar bulk density after drying. However bentonite has a typical property. When water is added to bentonite it disintegrates in to smaller particles with high surface area. On drying these particles provide good adhesion to the other aggregates and provides more shrinkage in the body. Thus the samples prepared with more amount of bentonite have slightly more bulk density. BD of all the samples

increases as a function of temperature. This is attributed to the densification of the sample which is quite obvious. It could also be seen that change in BD as a function of increase in temperature increases. This is attributed to enhance liquid phase formation at high temperature leading to enhance densification of the sample.

4.1.6 Cold crushing strength of 45% alumina mortar prepared with bentonite binder

Cold crushing strength of the mortar as a function of temperature and amount of bentonite has been shown in Fig.4.5.

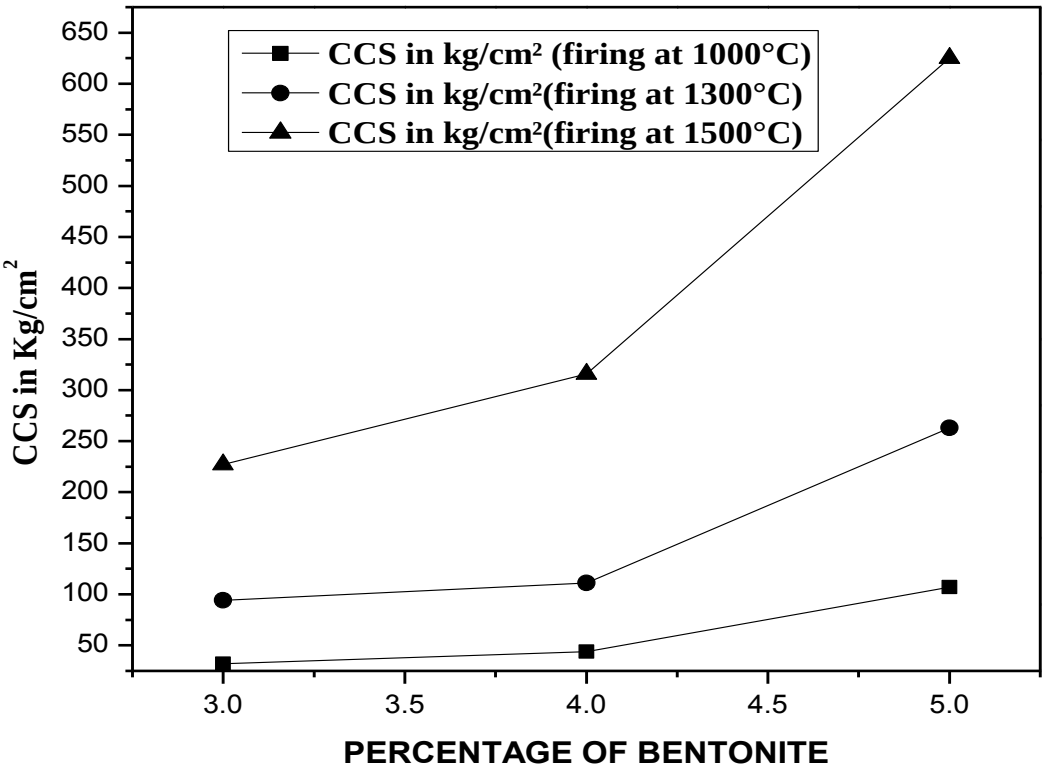


Fig.4.5. cold crushing strength of 45% alumina mortar as a function of bentonite and sintering temperature

It could be seen from the figure that the CCS value of the samples increases with bentonite content. Bentonite on addition of water disintegrates in to smaller particles with high surface area. These smaller particles provide good adhesion with other constituents of the aggregates and also provide more shrinkage in the final body. This adhesion increases with increase in bentonite content in the composition. Thus the sample prepared with 5% bentonite shows

high CCS value for all the temperature studied. The CCS of all the samples increases with increase in temperature. This is correlated with the increase in bulk density or decrease in porosity of the sample. As the firing temperature increases more and more densification occurs in the sample. As a result, the contact area between the aggregates increases. This gives more strength in the sample with increase in firing temperature. The rate of increase of CCS value as a function of increase in temperature is found to be more. This phenomenon is attributed to the enhanced densification of the sample at high temperature due to formation of more liquid phases.

4.1.7 Cold modulus of rapture of 45% alumina mortar prepared with bentonite binder

Cold modulus of rapture of the mortar as a function of temperature and amount of bentonite has been shown in Fig. 4.6.

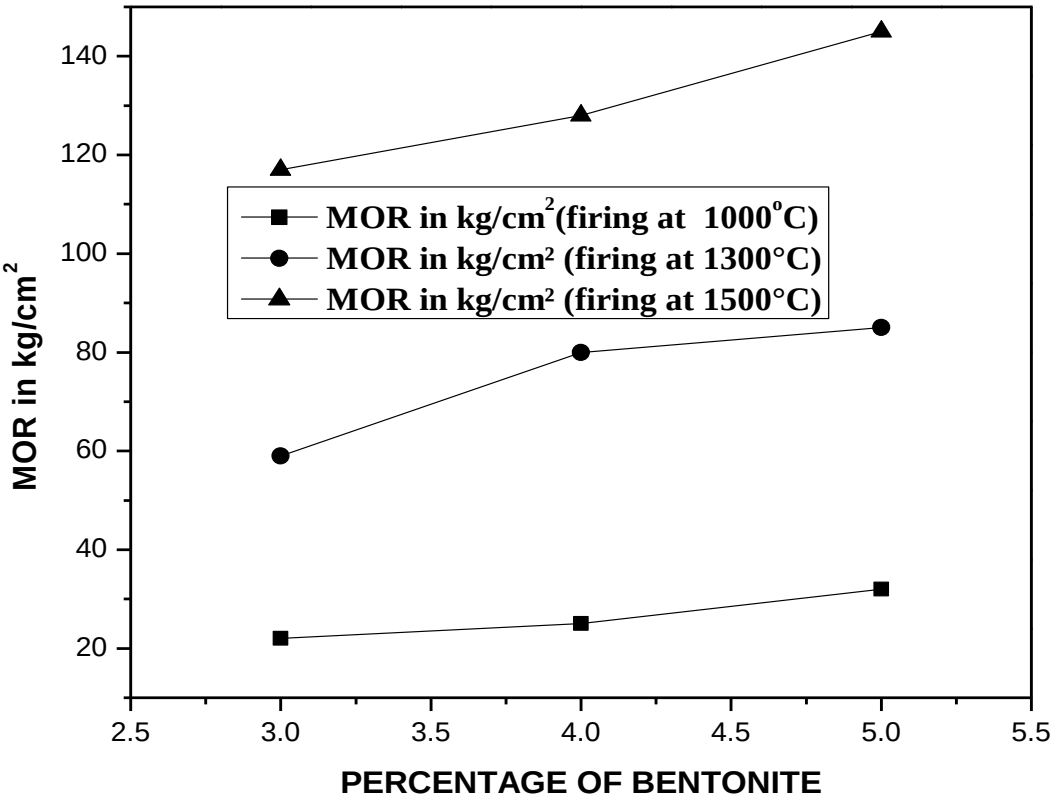


Fig. 4.6 Modulus of rupture of 45% alumina mortar as a function of bentonite and sintering temperature

It could be seen from the figure that MOR of the sample increases with increase in bentonite content at a particular temperature. Similar behaviour is observed for all the temperature studied. Bentonite on addition of water disintegrates in to smaller particle with high surface area. This high surface area particle provides strong adhesion with the aggregates. Thus the samples prepared with 5% bentonite showed more MOR value as compared to that prepared with 3% bentonite at a particular temperature. The MOR value of the sample increases with increase in sintering temperature and is attributed to the densification of the sample with increase in sintering temperature. MOR value of the sample prepared with 5% bentonite was fund to decrease as compared to that prepared with lower amount of bentonite when sintered at high temperature. Bentonite is well known be associated with more alkalies like sodium or calcium. As the bentonite content increases the alkali content in the sample also increases this leads to formation alkali-alumino silicate glass in the fired samples. The increase in glassy phase in the sample is correlated to the decrease in MOR value of the sample.

4.1.8 Bond Strength of 45% alumina mortar prepared with bentonite binder

Bond strength of 45% alumina mortar as a function of bentonite content and temperature has been shown in Fig. 4.7.

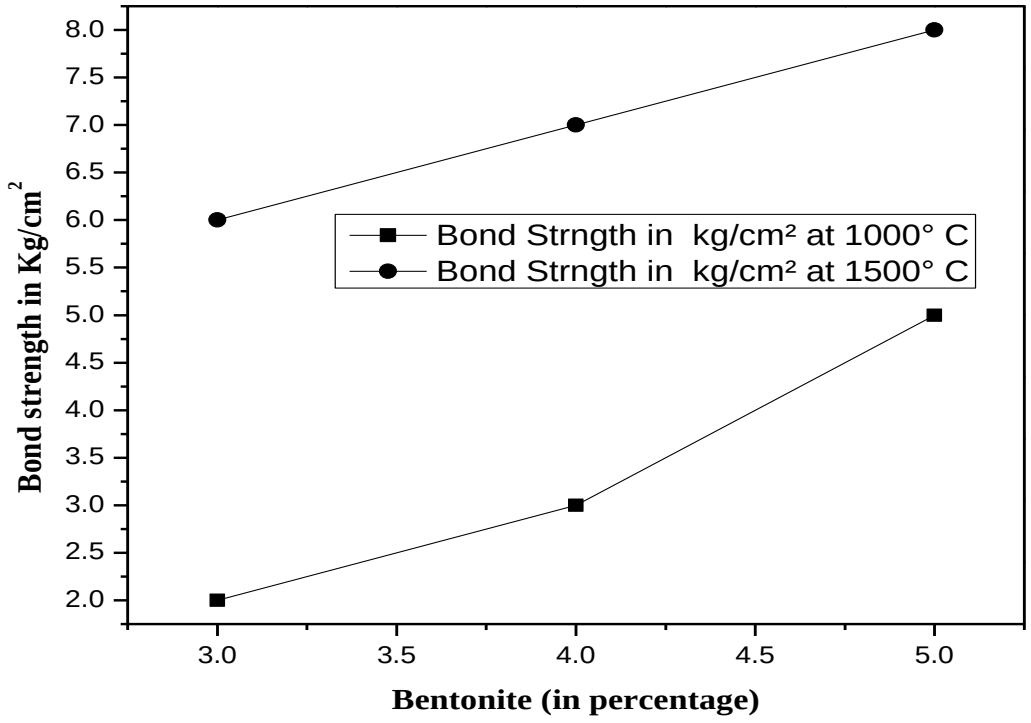
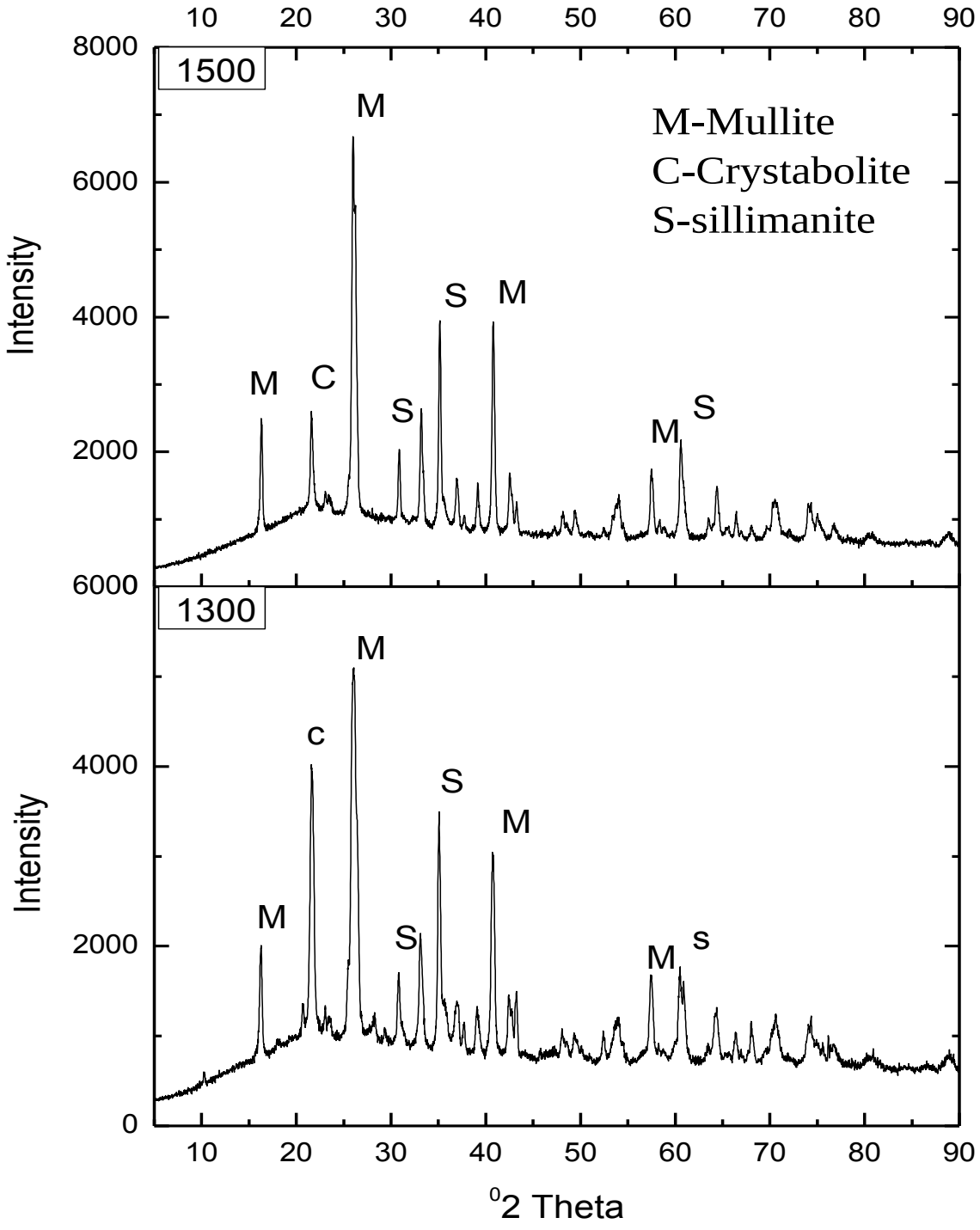


Fig. 4.7 Bond strength of 45% alumina mortar as a function of bentonite and sintering temperature

It could be seen from the above figure that bond strength value of the sample increases as a function of bentonite content and temperature. When two half bricks are joint together by the help of this mortar, at lower temperature (1000^0C) there is no liquid formation. Bonding between mortar and brick is only due to solid phase sintering which does not provide more strength to the joint. Thus at lower temperature bond strength of the mortar joint is very less. As the firing temperature increases to 1500^0C, more densification occurs in mortar sample which leads to the formation of more amounts of liquid phases in the mortar. Bonding of brick mortar is due to this liquid phase sintering. This liquid phase created in the sample mortar fills the voids or pores present in the brick. Thus, joint strength of the brick to mortar joints increases. As previously discussed, bentonite has a good adhesion properties. In addition of water it disintegrates in to smaller particles which give high surface area to adhere the other constituents of the aggregates. This gives high strength to the mortar. This adhesion property of bentonite increases with the amount of bentonite added in to formulation. Thus, 5% bentonite content showed more bond strength as compared to 3% bentonite. The rate of increase of bond strength value of the mortar joint while joining two bricks as a function of content of bentonite and also as a function of temperature is found to be more. This phenomenon is attributed to the enhanced densification of the sample at higher temperature which increases the liquid phase formation.

4.1.9 Phase analysis sintered 45% alumina mortar prepared with bentonite binder

XRD patterns of 45% alumina mortar prepared with 4% bentonite as a function of firing temperature has been shown in the Fig. 4.8. It could be seen from the above XRD pattern that, the major phases are found in sample fired at 1300^0C and 1500^0C are mullite, sillimanite and crystabolite. While increase in firing temperature, it could be observed from the XRD figure that intensity of mullite peak increases while the intensity of sillimanite and crystabolite peak decreases with increase in firing temperature. All the samples prepared with different amount of bentonite shows similar behaviour. It could be concluded from the above data that, the liquid formation at higher temperature is the major cause behind the increasing mullitisation of the product. This increase in mullite content with increase in temperature plays a major role to enhance the mechanical behaviour of the product. This analysis also showed that, at a particular temperature with different content of bentonite, mullitization of the product increases. The product having more bentonite content showed better mechanical properties as compared to other having less bentonite content.



[Fig. 4.8 XRD patterns of sintered 45% alumina mortar prepared with bentonite]

4.2 Effect of plastic clay on the Properties of 45% alumina based alumina-silicate mortar:

4.2.1 Batch formulation of 45% Al_2O_3 mortar prepared with plastic clay binder

Raw materials used in the sample preparation of 45% alumina based alumino-silicate mortar are same as in the previous section. The effect of Plastic clay on the properties of mortar has been studied on the basis of linear shrinkage, strength and other properties.As from previous section it has been found that composition-2 containing 5% plastic clay is an optimum amount which gives best properties at high temperature also at low temperature. Mortar was prepared with this composition (T2) with varying plastic clay content. In the present study, four different formulations have been prepared by varying plastic clay. Batch formulation of mortar has been shown in Table IV.II.

Table IV.II: Batch formulation of 45% Al_2O_3 mortar prepared with plastic clay binder

Composition (%)	T1	T2	T3	T4
Fire clay grog (-200 mesh)	40	35	30	25
Silliminite sand (-200 mesh)	20	20	20	20
Chinese Bauxite (-200 mesh)	20	20	20	20
Bentonite	5	5	5	5
Plastic clay	15	20	25	30

4.2.2 Flow properties of 45% Al_2O_3 mortar prepared with plastic clay binder

Flow behaviour of the mortar as a function of plastic clay has been graphically presented in Fig. 4.9. It could be observed that, mortar prepared with T4 showed highest flow value around 39% and that prepared with T1 showed lowest flow value around 18%, whereas the mortars prepared with T3 and T4 showed almost similar value. Flow behaviour of all the formulations has been studied by adding 40% water to it. Flow of a mortar depends mostly on the binders and plasticizers.

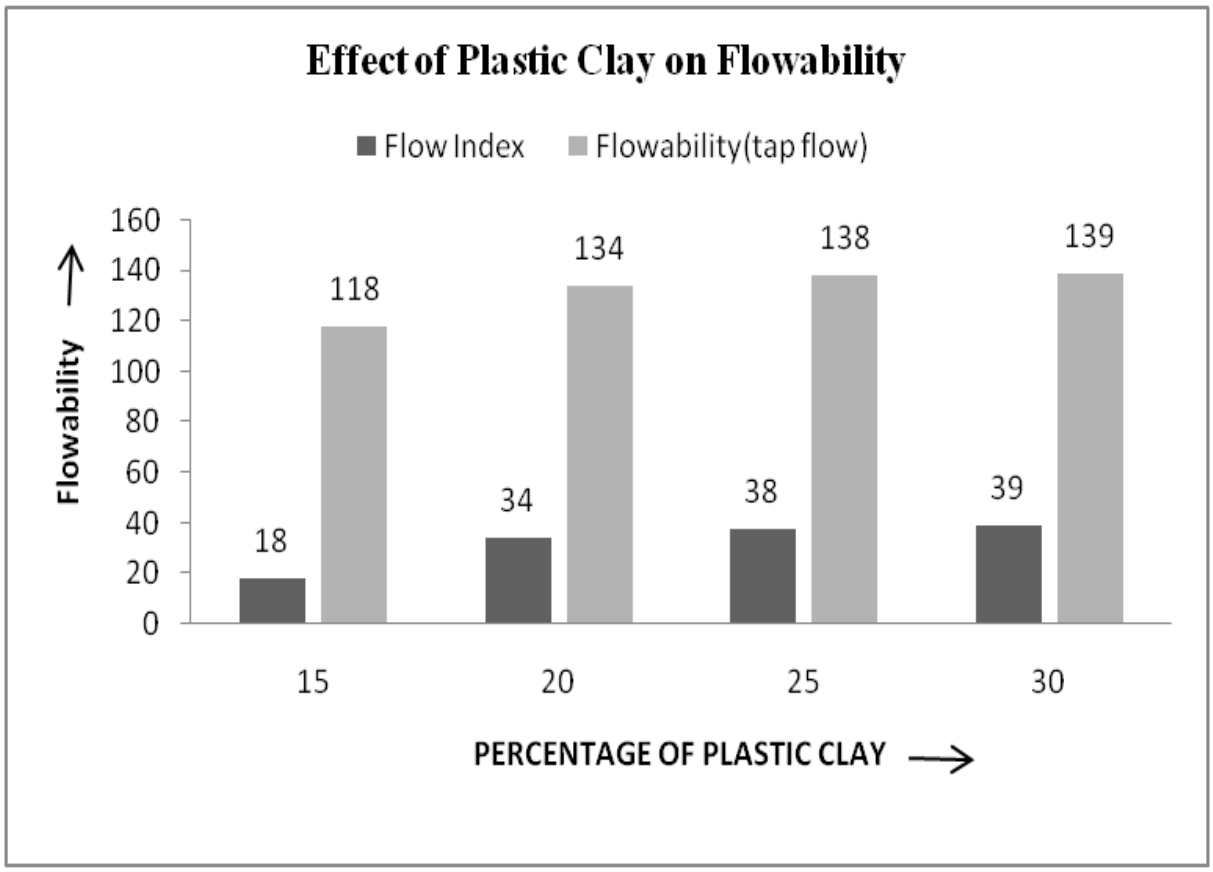
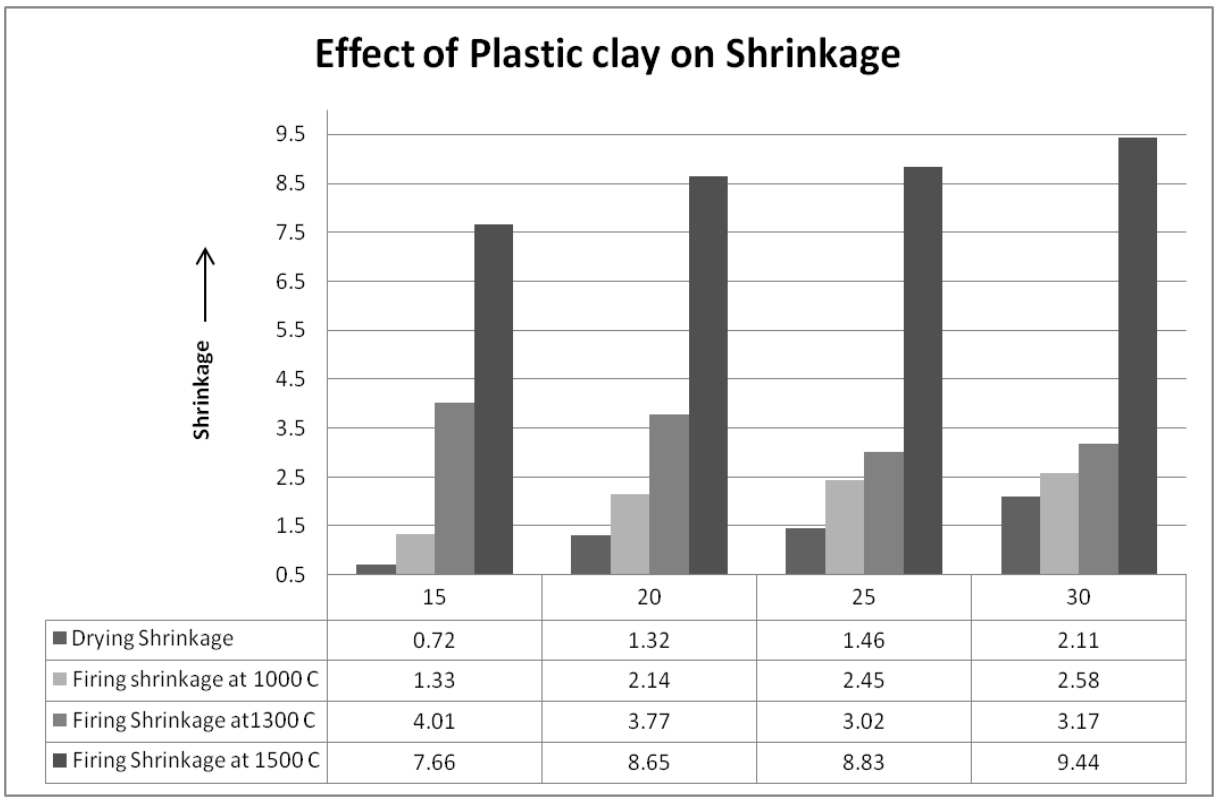


Fig.4.9. Flow Behaviour of 45% alumina mortar as a function of Plastic Clay and sintering temperature

Water used to incorporate flow behaviour of the mortar is utilized in two ways. One part of the water is utilized to fill up the interstitial porosity of the mix formed by the aggregates and binders and the other part imparts the flow behaviour of the mortar. The more amount of water is available for second purpose; the mortar formation will show more flowability. Plastic clay used in the formulation has fine particle size as compared to the other constituents. Plastic clay used in the formulation act as filler owing to its fine particle size. Thus plastic clay fills up the interstitial pores formed by the aggregates and improves packing density of the structure. Hence the increase in plastic clay amount in the body formulation increases particle packing density and it leads to less void space present in the mortar structure. Thus a mortar structure prepared with high amount of plastic clay has less porosity. Amount of water needed to fill up inter aggregates porosity decreases and more amount of water are available to impart flowability for a mortar prepared with more amount of plastic clay. Thus, flowability of mortar increases with increase in percentage of plastic clay in the body formulation. T4 composition shows high flowability owing to high plastic clay content.

4.2.3 Shrinkage behaviour of 45% Al_2O_3 mortar prepared with plastic clay binder

Shrinkage behaviour of the mortar as a function of temperature and percentage of plastic clay has been shown in Fig.4.10. It could be seen from the figure that the drying shrinkage increases with increase in plastic clay content of the body formulation. Shrinkage also found to increase with increase firing temperature for all the formulation studied. Firing shrinkage at a particular temperature was found to be more for the samples prepared with more amount of plastic clay.



	15	20	25	30
Drying Shrinkage	0.72	1.32	1.46	2.11
Firing shrinkage at 1000 C	1.33	2.14	2.45	2.58
Firing Shrinkage at1300 C	4.01	3.77	3.02	3.17
Firing Shrinkage at 1500 C	7.66	8.65	8.83	9.44

Fig.4.10. Shrinkage behaviour of 45% Al_2O_3 mortar as a function of plastic clay and temperature

It has been discussed earlier that the mortar prepared with more amount of plastic clay has more flowability. This indicates that a quite substantial amount of liquid film formation around the particles with in turns increases the flowability of the mortar. The samples with more the thicknesses of the liquid film formed are liable to have more drying shrinkage. Thus, the samples prepared with more amount of plastic clay showed high drying shrinkage. The increase in firing shrinkage with increase in firing temperature for a particular formulation is attributed to the increase in density of the sample with increase in temperature which is quite obvious. The shrinkage rate was found to increase with increase in firing

temperature for all the composition studied. This phenomenon may be attributed with the liquid phase sintering of the mortar. Some amount of alkali present in the plastic clay which forms liquid phase at higher temperature of firing. The amount of liquid phase increases with increase in temperature leading to enhanced densification rate of the sample. Thus the shrinkage rate increases with increase in firing temperature. Shrinkage with increase in plastic clay content at a particular temperature was found to increase with increase in plastic clay amount. Plastic clay is the low melting constituents of all the components used in the body formulation. The above property is attributed to the formation of more liquids at high temperature with increase in plastic clay content in the sample.

4.2.4 Bulk density of 45% Al_2O_3 mortar prepared with plastic clay binder

Bulk density of the mortar as a function of temperature and percentage of plastic clay has been shown in Fig. 4.11. It could be seen from above graph that, bulk density of mortar increases from T1 to T3 after that it remains constant as a function of temperature and plastic clay content. Liquid phase formed at higher temperature as plastic clay contains alkali. This liquid phase amount increases with increase in plastic clay content of the formulation as well as increase in temperature. The liquid phase helps in liquid phase sintering of the mortar and increase the bulk density of the sample. The increase in bulk density in the low temperature range (1000^{o}C) is quite small and is attributed to the neck growth in the sample due to solid state sintering. The rate of change of bulk density was found to increase with increase in firing temperature and is attributed to the enhanced densification with the increase in liquid phase formation.

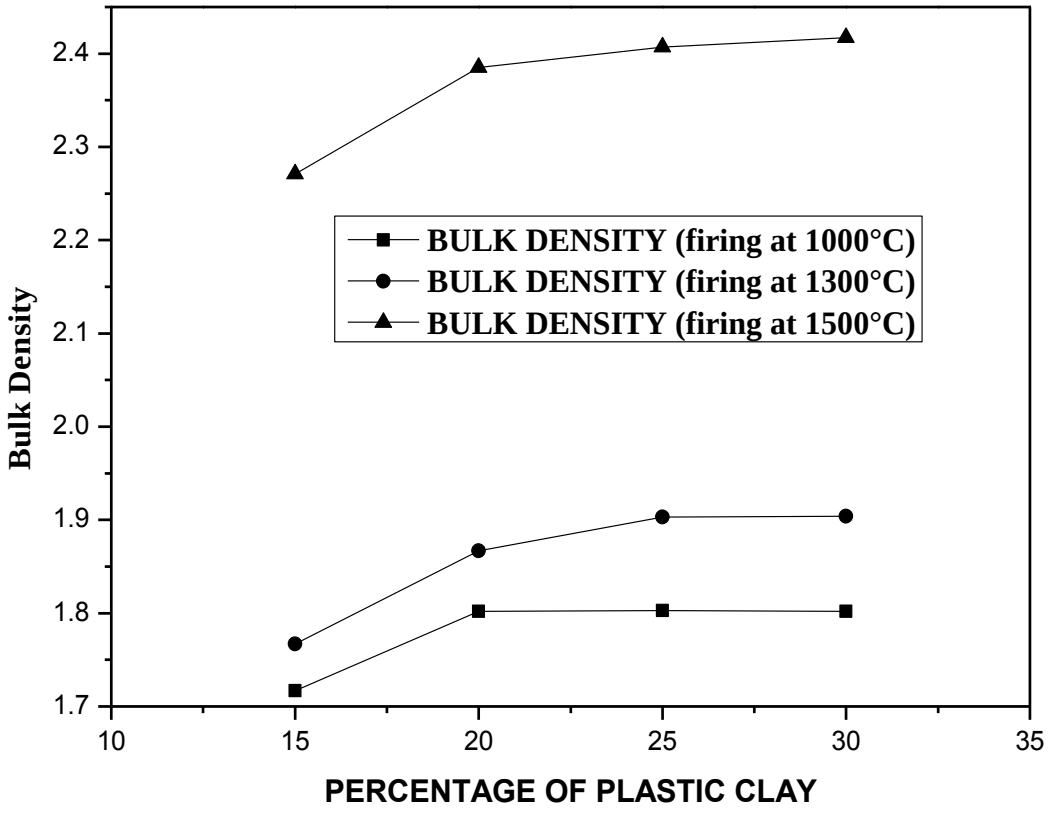


Fig.4.11. Bulk Density of 45% Al_2O_3 mortar as a function of plastic clay

4.2.5 Apparent porosity of 45% Al_2O_3 mortar prepared with plastic clay binder

Apparent porosity of the mortar as a function of temperature and plastic clay content has been shown in Fig. 4.12. It could be seen from above graph that, with increase in temperature and plastic clay content apparent porosity of the sample follows an inverse relationship as compared with bulk density. Apparent porosity of the prepared samples decreases as a function of plastic clay content and also with temperature. It has also been found that after a certain amount of plastic clay content it remains constant. As discussed earlier, plastic clay contains alkali which gives rise to liquid phases at higher temperature. This liquid phase amount increases with increase in plastic clay content of the formulation as well as increase in temperature. This liquid phase helps in liquid phase sintering of the sample. Thus apparent porosity of the sample decreases at higher temperature firing as well as at higher amount of plastic clay. Above graph also shows that apparent porosity of sample firing at 1300^0C and 1500^0Cdecreases with increase in temperature and also increase in plastic clay content due to high liquid phase formation.

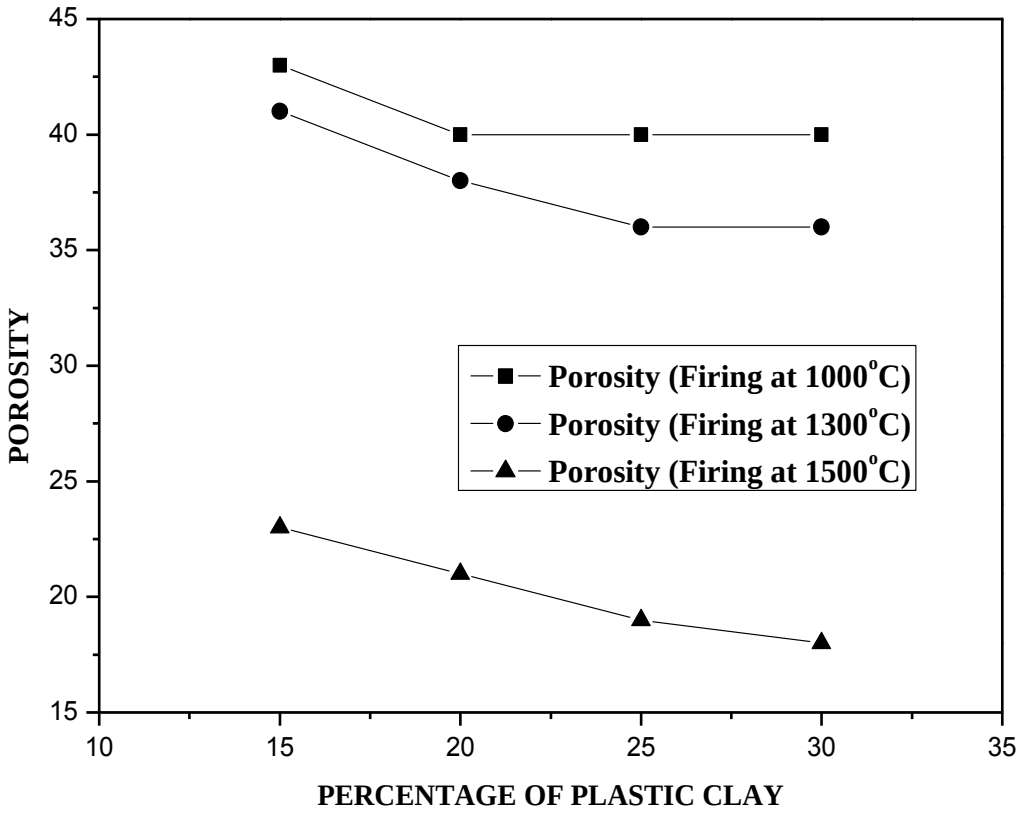


Fig.4.12. Porosity of 45% Al_2O_3 mortar as a function of plastic clay

It could be also found that at a particular temperature porosity decreases with increase in plastic clay content in the mortar composition. This is attributed to the packing density of the mortar. Plastic clay has fine particle size as compared to the other constituent of the mortar. It acts as filler and fills the interstitial voids of the aggregates. Thus with increase in plastic clay content of the mortar more and more interstitial voids of the aggregate fills up leading to higher packing density of the sample. Thus the sample prepared with 15% plastic clay shows more apparent porosity as compared to that prepared with 30% of plastic clay content. At a particular composition Porosity decreases as a function temperature. When temperature of a sample having a particular plastic clay content increases liquid formation due to alkali content of plastic clay also increases as a function temperature which leads to more liquid phase sintering at higher temperature. This liquid phase sintering packs the particles more and more which decreases the porosity. Porosity of all the samples irrespective of composition showed a higher porosity (15%-30%) as compared to that fired at 1500^0**C.** At low

temperature plastic clay does not produce any liquid phase so the porosity at this point is mostly due to the packing of the particles. A small decrease in porosity occurs due to solid state sintering. Rate of decrease of porosity with increase in temperature will enhance the rate of densification of the sample.

4.2.6 Cold Crushing Strength of 45% Al_2O_3 mortar prepared with plastic clay binder

Clay minerals are composed of fine particles of hydrous alumina-silicates which develop plasticity when they are mixed with water [4]. Cold crushing strength of the mortar as a function of temperature and percentage of plastic clay has been shown in Fig. 4.13.

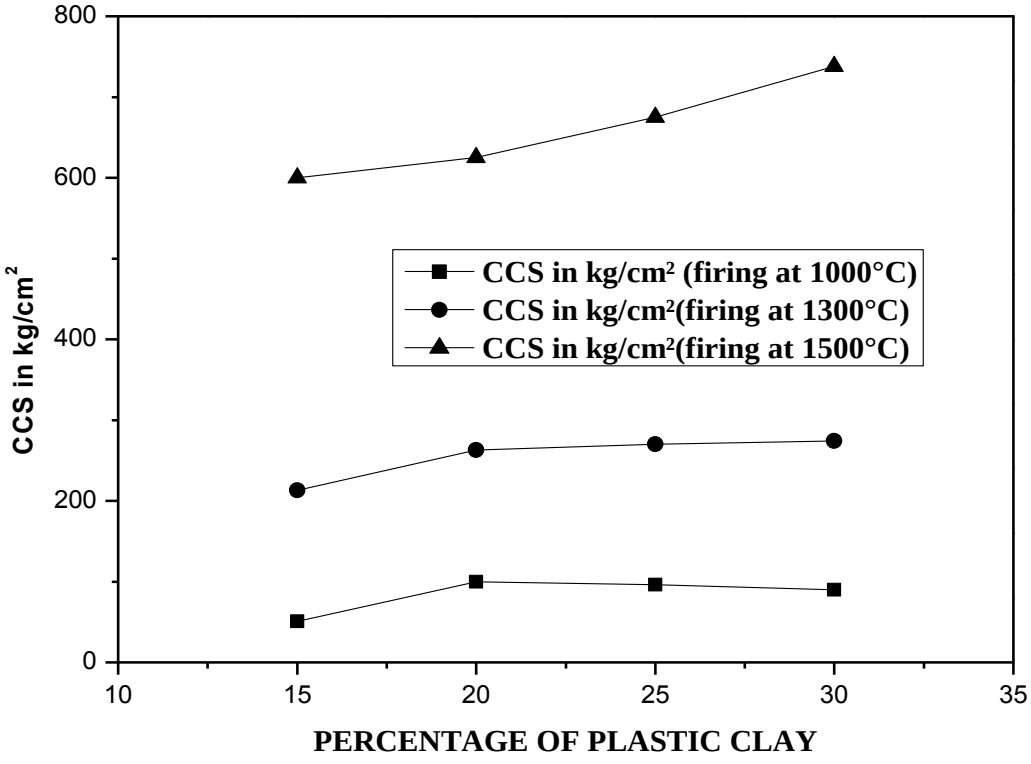


Fig.4.13. Cold Crushing Strength of 45% Al_2O_3 mortar as a function of plastic clay and temperature

For all the temperature studied, CCS increases with increase in plastic clay content. This is correlated with the increase in density of the sample with increase in plastic clay content in the composition. As discussed earlier, plastic clay acts as a filler and fills the interstitial voids of the aggregate. Thus increase in plastic clay content in the composition leads to more packing density of the sample. More the packing density better will be the fired density. Thus the sample prepared with 15% plastic clay showed lower bulk density as compared to that of

30% sample. The more the porosity in the sample lower will be its cold crushing strength. Hence the sample prepared with 15% plastic clay showed lower CCS value as compared to 30%.Plastic clay contains alkali phase.At a particular composition, as plastic clay contains alkali phases it creates liquid phase at higher temperature. This Liquid phase formationgradually increases for a particular composition with increase in temperature. This liquid phase increases the packing of the sample by liquid phase sintering which leads to increase in CCS value. At 1500^0C more amount of liquid formation takes place due to which increase in CCS value for changing in temperature at 1500^0C is more as compared to other temperature. This is due to more mullitization at highest temperature. Rate of change of CCS value with increase in firing temperature increases which is attributed to the densification of the sample.

4.2.7 Cold Modulus of Rapture of 45% Al_2O_3 mortar prepared with plastic clay binder

Cold modulus of rapture of the mortar as a function of temperature and percentage of plastic clay has been shown in Fig.4.14.

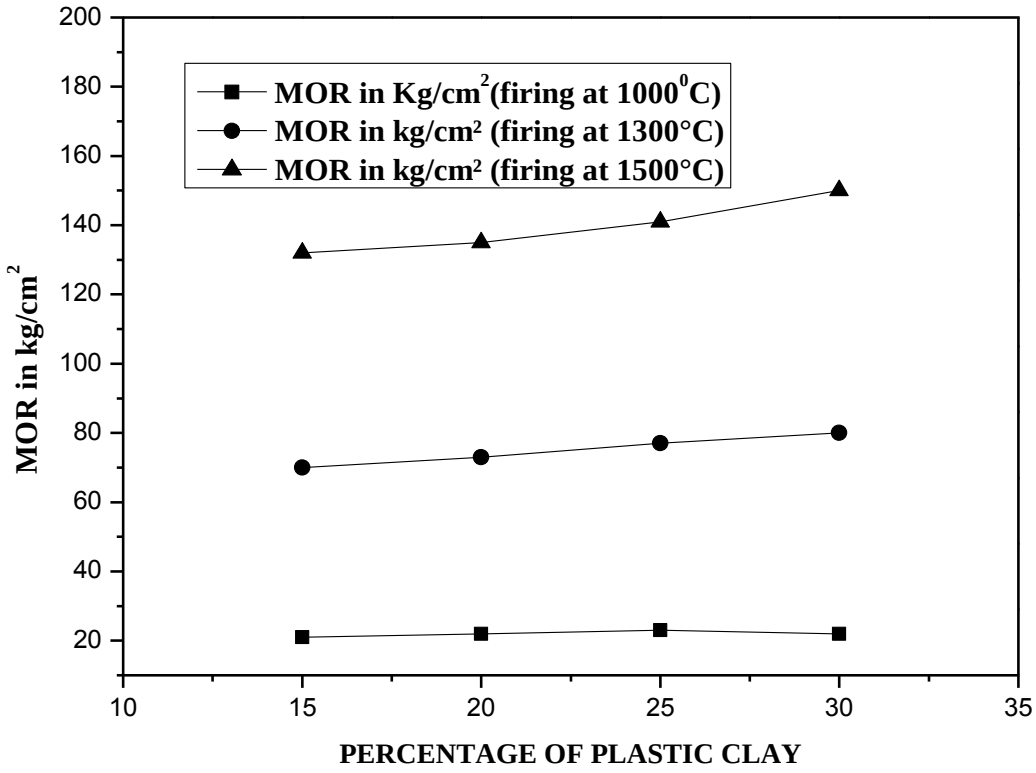


Fig.4.14. Modulus of Rupture of 45% Al_2O_3 mortar as a function of plastic clay

Modulus of rupture of a sample increases as a function of plastic clay content and also with temperature. It could be seen from the above graph that MOR of the sample increases as a function of temperature at a constant rate at a particular composition. At a particular temperature MOR slightly changed with increase in plastic clay content. As previously explained, density of the sample increases with increase in plastic clay content and as well as increase in temperature. This increase in density decreases the voids present in the aggregate. These voids are filled up by plastic clay content which decreases the porosity of the sample. Thus, increase in plastic clay content increases the density of the sample at lower temperature also at higher temperature which leads to increase in CCS value. More the CCS value of the sample higher is the MOR value of the sample.

4.2.8 Bonding Strength of 45% Al_2O_3 mortar prepared with plastic clay binder

Understanding bond strength properties is essential in the design and construction of brick walls. Inadequate bond strength can lead to cracking in masonry unit construction which can cause extensive damage because it is a brittle mode of failure. Anticipating failure due to inadequate bond strength is difficult to predict visually and could potentially lead to widespread damage, and eventually lead to catastrophic failure if a high force or load is applied. The forces of adhesion and cohesion make up bond strength. Forces attracting mortar to it is called cohesion and forces attracting mortar to the brick surface is adhesion. Strength of mortar strongly depends upon binder used in the mortar, its packing with the aggregates [46].

Bonding Strength of the mortar as a function of temperature and plastic clay percentage has been shown in Fig.4.15. It could be seen from the above graph that bond strength of the mortar while joining two half bricks together increases with increase in plastic clay content as well as in temperature. It could also be seen that at a particular temperature due to increase in plastic clay content bond strength of the sample increases with increase in density of the sample. Rate of change of bond strength increases with increase in the firing temperature of the sample, at particular composition bond strength of a sample also increases as a function of temperature. This is due to more liquid phase formation in the sample.Plastic clay due to its fine grain size fills up all the interstitial spaces or voids which increases its density at lower temperature. At higher temperature plastic clay produce liquid phase during densification of the sample which increase density of the sample at higher temperature. More plastic clay content will enhance the above two process which increases the density of the sample more and more due to which bond strength of the mortar increases.

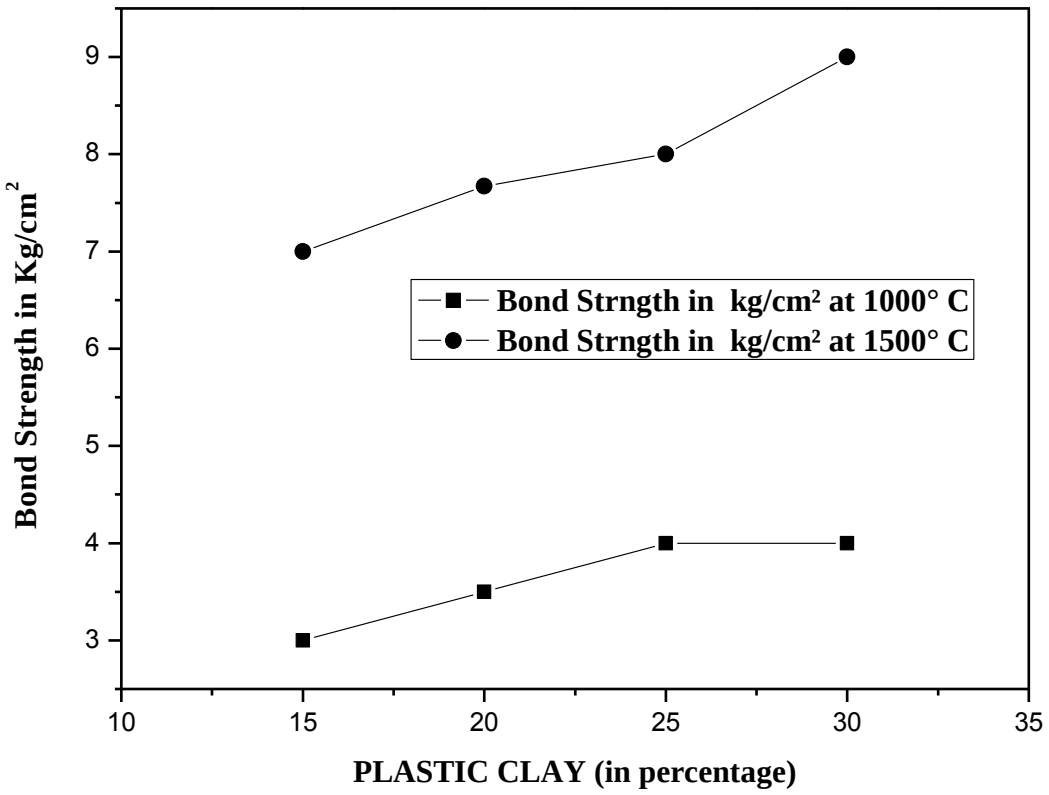
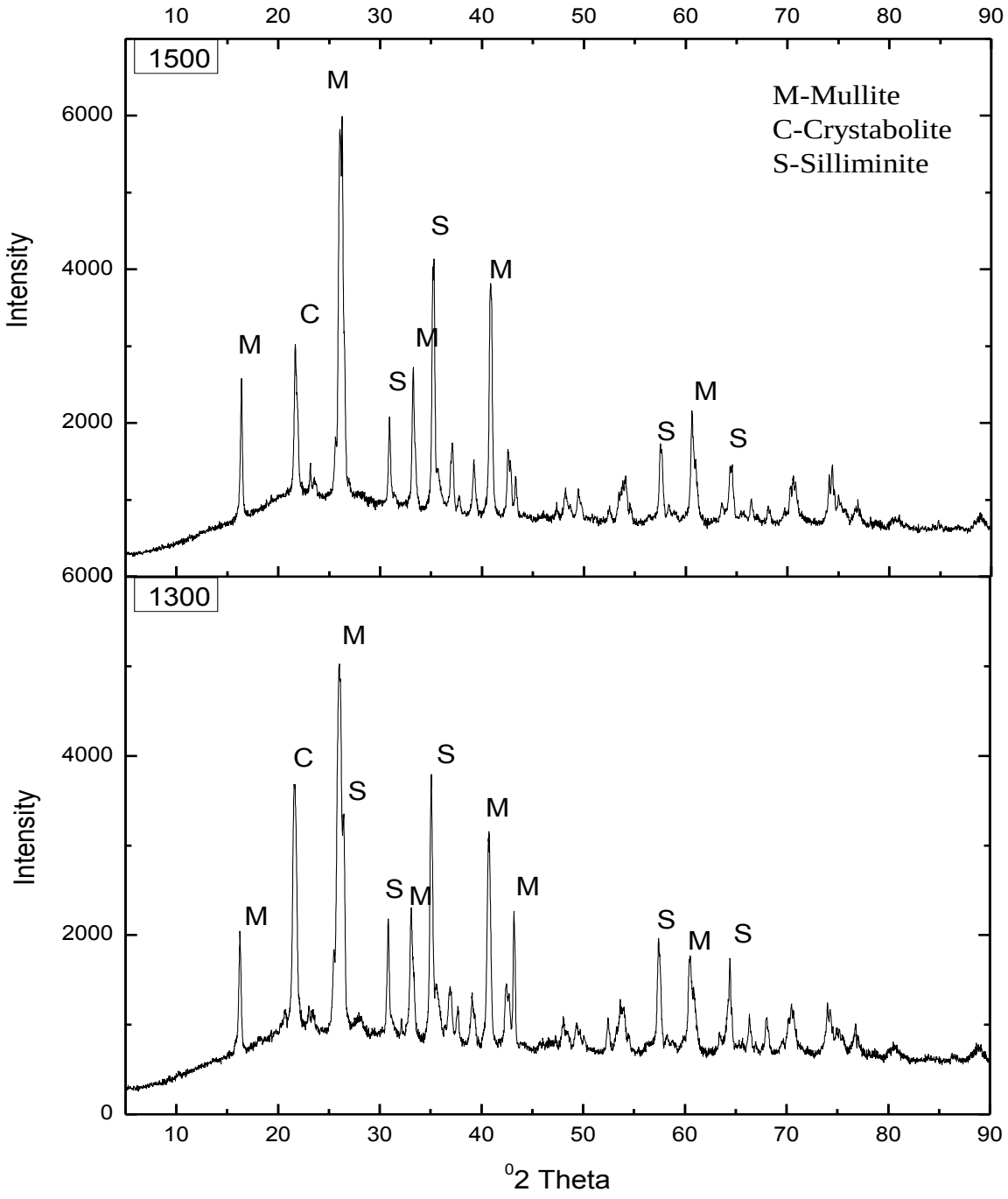


Fig.4.15. Bond Strength of 45% Al_2O_3 mortar as a function of plastic clay

4.2.9 Phase analysis of 45% Al_2O_3 mortar prepared with plastic clay binder

XRD patterns of Composition T2 (45% Al_2O_3) mortar fired at different temperature 1300 °C and 1500°C has been shown in the Fig.4.16. The entire sample prepared with different amount of plastic clay content showed the similar behaviour. It could be seen from the XRD patterns that sillimanite, mullite and crystabolite are the major phases in the sample fired at 1300^0C and 1500^0C.a closer look on the patterns reveals that the intensity of mullite peak increase while the crystabolite and sillimanite decreases with increase in firing temperature. Thus it could be concluded that the liquid form at high temperature may have a roll in increasing mullitization of the product. This increase in mullite content of sample with increase in temperature plays an important role in enhancing the mechanical behaviour of the product. Analysis of the XRD pattern as afunction of plastic clay content at a particular

temperature showed more mullitisation with increase in plastic clay. Thus the samples prepared with more plastic clay showed enhance mechanical behaviour.


1500
1300
M-Mullite
C-Crystabolite
S-Silliminite
Intensity
Intensity
0
2000
4000
6000
10
20
30
40
50
60
70
80
90
M
C
S
M
S
M
S
M

4.3 Batch formulation of 45% Al_2O_3 mortar with a new binder named sodium silicate in different percentage:

Raw materials used in the body formulations of 45% Al_2O_3 mortar are same as in previous sections. Mortar was prepared with same composition with varying sodium silicate binder percentage and effect of binder on the properties of mortar has been studied. Batch formulation of mortar has been given in Table IV.III.

TABLE IV.III Batch formulation of 45% Al_2O_3 mortar prepared with sodium silicate liquid binder

Composition	S1	S2	S3	S4
Fire clay grog (-200 mesh)	35	35	35	35
Silliminite sand (-200 mesh)	20	20	20	20
Chinese Bauxite (-200 mesh)	20	20	20	20
Bentonite	5	5	5	5
Plastic clay	20	20	20	20
Sodium silicate liquid (soda:silica-1:3)	10	15	20	25
Water Content (in%)	20	15	10	5

4.3.1 Flow properties of 45% alumina mortar prepared with sodium silicate liquid binder

Variation flowability to the mortar composition as a function of liquid sodium silicate percentage has been studied and graphically presented in Fig.4.17. It could be seen from the figure that the flowability of the mortar increases with increase in sodium silicate content in the mortar. The amount of total water in all the compositions studied was constant.

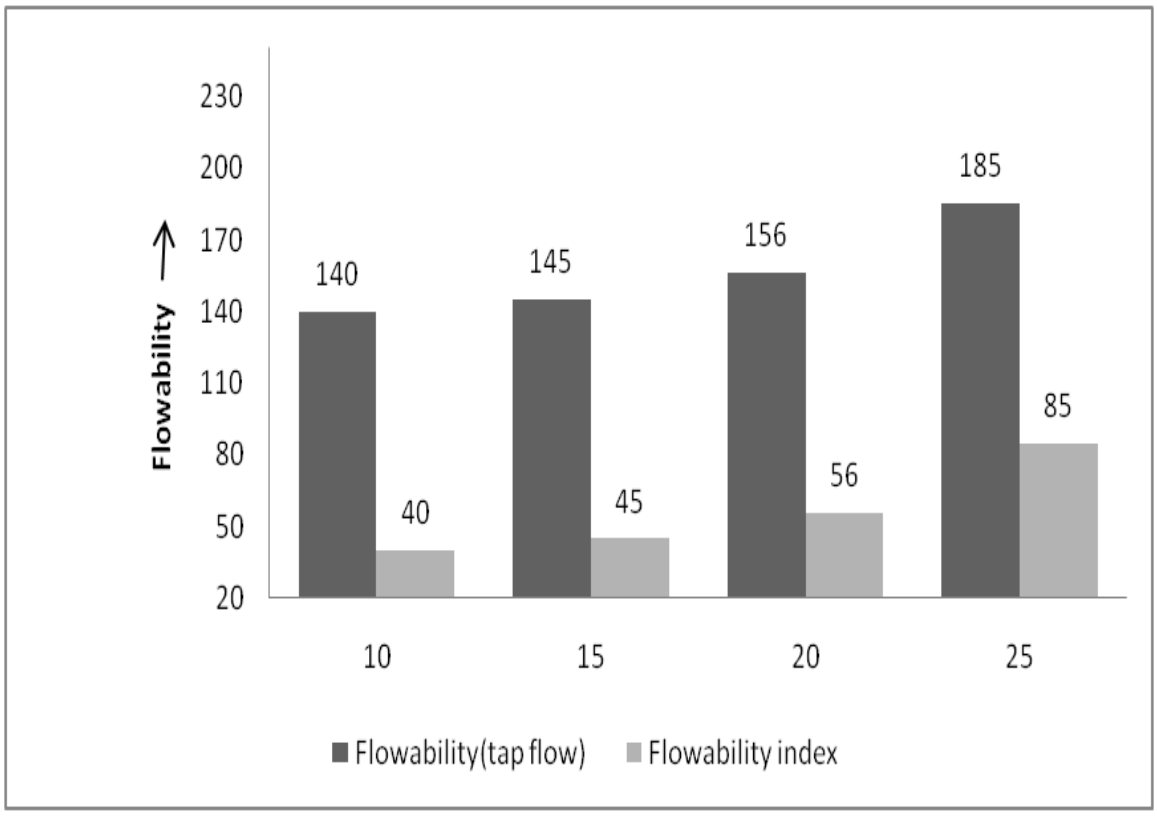


Fig.4.17. Flowability of 45% Al_2O_3 mortar as a function of sodium silicate liquid content and temperature

The only difference of the mortar formulation lies with the increase in sodium silicate content added in the form of liquid. Sodium silicate is well known as deflocculent in ceramic system. It alters the surface of clay particles used in the body formulation and thereby decrease the viscosity of mortar paste. Thus with increase in sodium silicate in the body formulation the flowability of the mortar paste increases. Hence the sample prepared with 25% liquid sodium silicate showed high flowability.

4.3.2 Shrinkage Behaviour of 45% alumina mortar prepared with sodium silicate liquid binder

Drying and firing shrinkage of the refractory mortar as function of sodium silicate content is shown in Fig.4.18. It could be seen from the figure that shrinkage increases up to 20% liquid sodium silicate liquid addition there after it decreases for all the temperature studied. The sample prepared with 25% sodium silicate liquid showed less shrinkage as compared to that prepared with 20% sodium silicate liquid. Flowability of the mortar prepare with 25% sodium silicate liquid is more as compared with that prepared with 20% sodium silicate liquid. More

flowability of mortar results in better packing density due to rearrangement of aggregates as a result shrinkage value decreases. Shrinkage increases with increase in temperature. The increase in shrinkage with increase in temperature is attributed to the densification of the sample.

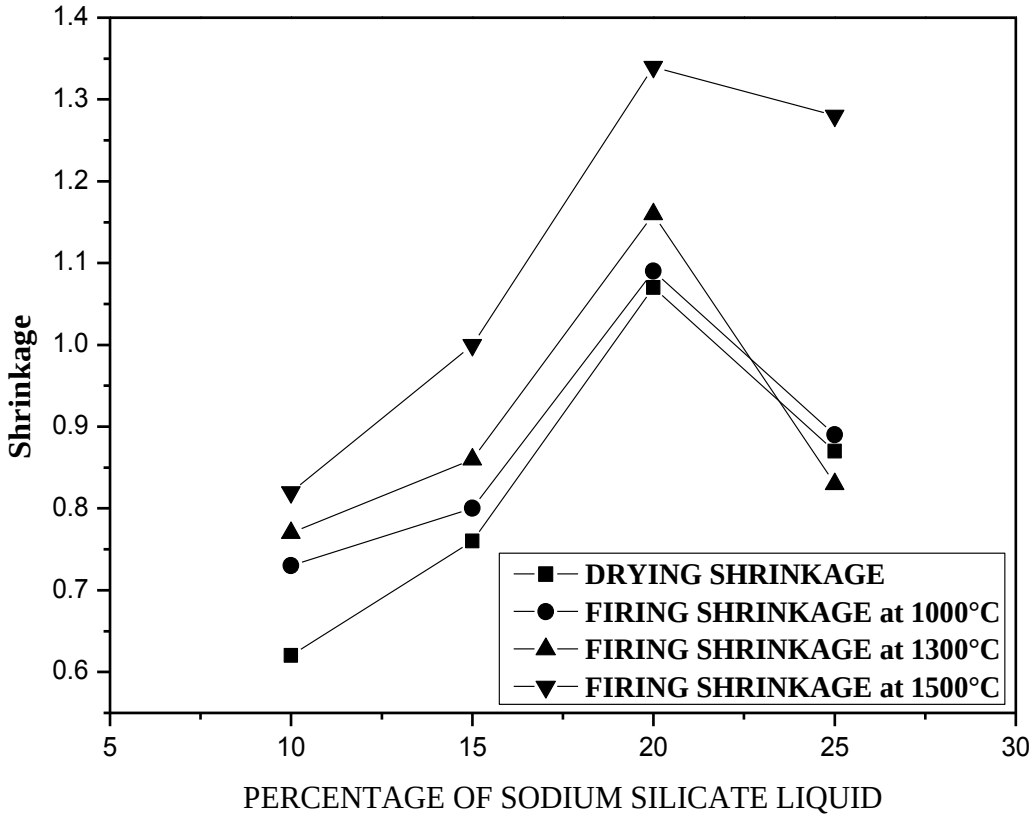


Fig.4.18. Shrinkage behaviour of 45% Al_2O_3 mortar as a function of sodium silicate liquid and temperature

4.3.3 Bulk Density of 45% alumina mortar prepared with sodium silicate liquid binder

Bulk density of the mortar sample as a function of sodium silicate content and temperature has been shown in Fig.4.19. It could be seen from the above figure that bulk density increases with increase in sodium silicate liquid content for all the temperature studied. With increase in sodium silicate liquid content, flowability of the mortar increases. Increase in flowability results in enhanced rearrangement of the aggregates leading to higher packing density of the mortar. The more the packing density the more will be the fired density. Thus the sample

prepared with more sodium silicate liquid content showed high bulk density. Bulk density increases with increase in temperature for all the composition studied due to the densification of the sample increases with temperature. Apparent porosity of the sample showed inverse relationship as that of the bulk density (not shown in the figure) which is quite obvious.

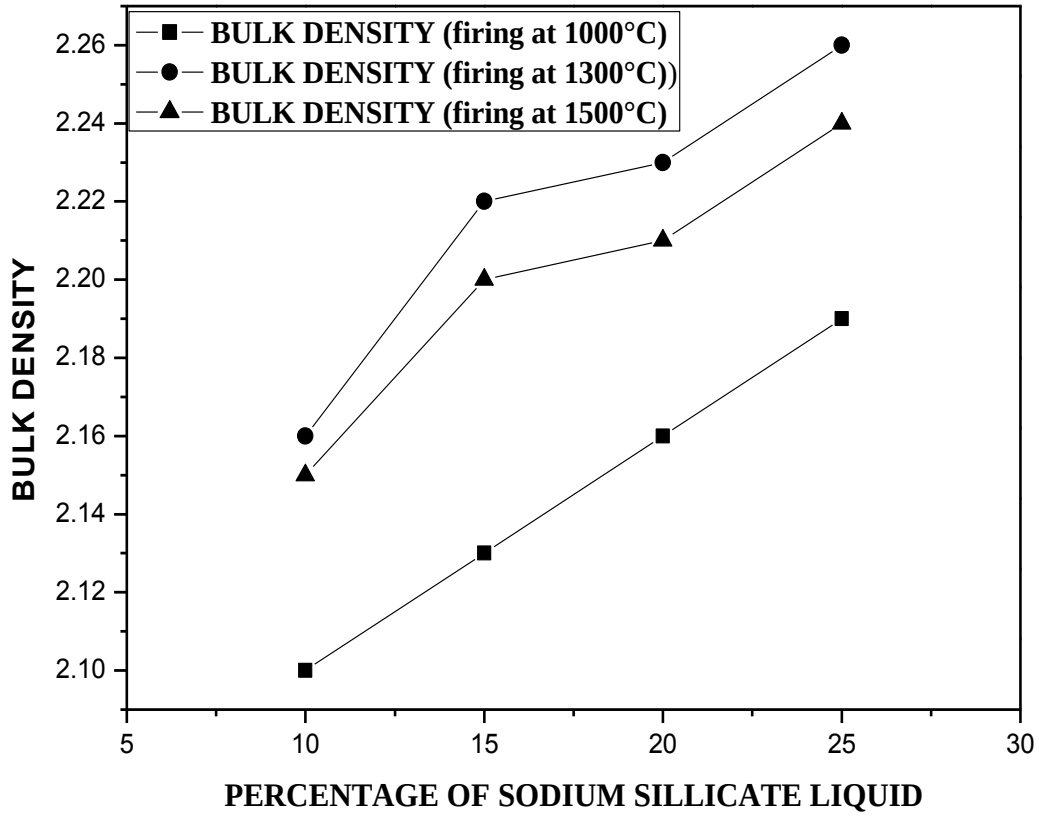


Fig.4.19. Bulk Density of 45% Al_2O_3 mortar as a function of sodium silicate liquid and temperature

4.3.4 Cold Crushing Strength of 45% alumina mortar prepared with sodium silicate liquid binder

Cold crushing strength of 45% Al_2O_3 mortar as a function of liquid sodium silicate content has been shown in Fig.4.20. It could be seen from the figure that at low temperature (1000^0C) cold crushing strength of the sample increases with increase in sodium silicate liquid content. In this low temperature firing substantial liquid phase will not occur which does not show substantial densification of the sample.

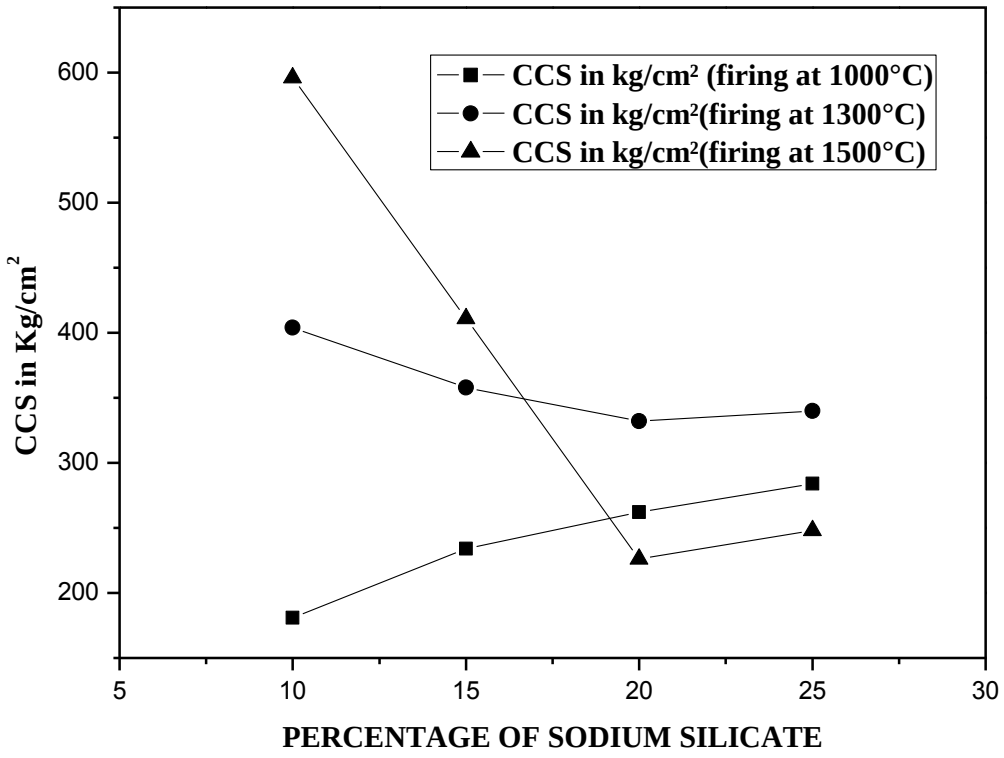


Fig.4.20. Cold Crushing Strength of 45% Al_2O_3 mortar as a function of sodium silicate liquid and temperature

So the increase in strength at this temperature as a function of sodium silicate liquid content is attributed to the deflocculation and cohesion behaviour of sodium silicate. At high temperature (1300^0C and 1500^0C) the cold crushing strength value of the sample decreases as a function of sodium silicate liquid content. The sodium silicate forms sodium alumina silicate glassy phase at higher temperature. These amounts of glassy phase formation increases with the increase in sintering temperature. The presence of glassy phase in the sintered body is correlated to the decrease in cold crushing strength value in the sample. The decrease in cold crushing strength value of the 1500^0C fired sample is quite substantial is correlated to the sodium alumina silicate glass formation.

4.3.5 Bond strength of 45% alumina mortar prepared with sodium silicate liquid binder

Sodium silicate is under go four distinct chemical reactions when applied as bond. These reactions have been defined as: hydration/dehydration, gelation, precipitation, surface charge

modification. These reactions allow silicate to act as a: film binder, matrix binder, chemical binder. Silicate can adhere an agglomerated material by one or more of its chemical reactions. Sometimes silicate-based formulations achieve their best performance by taking advantage of more than one of these adhesion mechanisms [34].

Effect of sodium silicate liquid upon bond strength of mortar has been studied and graphically represented in Fig.4.21.

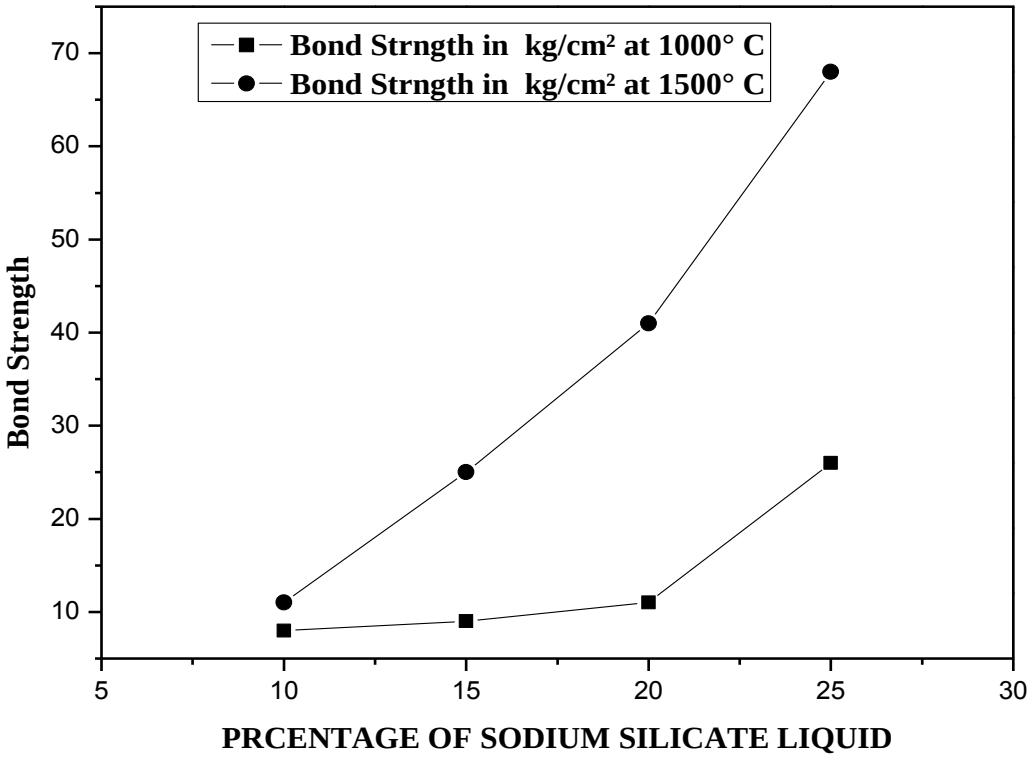


Fig.4.21. bond strength of 45% Al_2O_3 mortar as a function of sodium silicate liquid and temperature

It could be seen from the figure that bond strength of mortar increases with increase in temperature as well as with increase in sodium silicate content for all the samples. At higher temperature (1300^0C and 1500^0C) sodium silicate forms a glassy phase. This is due to the formation of sodium alumina silicate glass. This glassy substance penetrates in to the pores present in the brick surface which results a strong bond formation between the brick and mortar. This cocept is attributed to the bonding strength of the mortar. Thus, bonding strength of samples increases as a function of temperature. At lower temperature (1000^0C) glassy phase formation is less as compared to the sample fired at 1500^0C. Thus bond strength increases with increase in firing temperature. It could be also found from the figure that bond

strength increases with increase in sodium silicate liquid content. Increase in sodium silicate content increases the glassy phase formation at higher temperature which leads to increase in bonding strength of the mortar sample. When fired at higher temperature it forms glassier phase which binds aggregates material together to form a highly compact structure which increases its strength more and more. At lower temperature bond strength of the sample increases with increase in the sodium silicate liquid content. This is attributed to the deflocculation and cohesion behaviour of sodium silicate. Sodium silicate as binder gives equal strength amount at air drying which clay binder gives upon firing at lower temperature.

Chapter 5

Conclusion

Effect of bentonite on properties of alumino-silicate mortar:

The following conclusion is obtained from the study on the effect of bentonite content on the properties of the 45% alumina mortar:

- Flowability and tap flow index increases as a function of bentonite content. This is attributed to the hydration behavior of bentonite.
- Drying shrinkage of the mortar increases with decreases in bentonite content. This is attributed to the flowability of the mortar. Firing shrinkage of the mortar increases with increase in bentonite content and it is correlated to the densification of mortar due to low melting temperature of bentonite.
- Bulk Density of the sample increases slightly with increase in bentonite content in all the temperature. This is attributed to the densification and flow behavior of the sample.
- Room temperature mechanical behavior also increases as a function of bentonite content as well as temperature. This is correlated to the bulk density, porosity and densification of the sample respectively.
- Joint strength between brick and mortar increases only with increase in temperature. Mortar fired with lower temperature could not give required bond strength. This is correlated with densification of the sample at higher temperature.
- XRD study revealed enhanced mullitisation with increase in bentonite content as well as firing temperature and this enhance mullitisation which is correlated to the increase in mechanical property of the mortar.

Effect of plastic clay on properties of alumino-silicate mortar:

The following conclusion is obtained from the study on the effect of plastic clay content on the properties of the 45% alumina mortar:

- flowability (tap flow) and flowability index increases as a function of plastic clay content. This is attributed to the filling of interstitial voids of the aggregate by the plastic clay.

- Drying and firing shrinkage of the mortar increases with increase in plastic clay content and could be correlated with the flowability of the mortar.
- Bulk Density of the sample increases with increase in temperature as well as plastic clay content. This is attributed to the densification and flow behavior respectively.
- Room temperature mechanical behavior increases as a function of firing temperature as well as plastic clay content and is correlated to the bulk density, densification respectively.
- Joint strength between brick and mortar increases as a function of firing temperature. This is attributed to the behavior of the mortar at the firing temperature.
- XRD study revealed enhanced mullitisation with increase in plastic clay content as well as firing temperature and this enhance mullitisation which is correlated to the increase in mechanical property of the mortar.

Effect of sodium silicate liquid on properties of alumino-silicate mortar:

The following conclusion is obtained from the study on the effect of plastic clay content on the properties of the 45% alumina mortar:

- flowability (tap flow) and flowability index increases as a function of sodium silicate liquid content. This is attributed to the filling of interstitial voids of the aggregate by the sodium silicate liquid.
- Drying and firing shrinkage of the mortar increases with increase in sodium silicate liquid content for a certain extent after that it decreases. This is attributed with the flowability, packing density and densification respectively.
- Bulk Density of the sample increases with increase in temperature as well as sodium silicate liquid content. This is attributed to the densification and flow behavior respectively.
- Room temperature mechanical behavior increases as a function of sodium silicate liquid content only at lower temperature. At lower temperature it gives a reverse relationship with sodium silicate liquid content. This is correlated to the densification, sodium alumino-silicate glass formation respectively.

- Bond strength between brick and mortar increases as a function of firing temperature as well as sodium silicate liquid content. This is attributed to the deflocculation and cohesion behaviour, glassy phase formation at higher temperature by sodium silicate liquid.

Among all binders studied, sodium silicate was found to be more effective bond which increases the low temperature strength drastically; this is attributed to the air-setting behavior of the sodium silicate. However the mechanical properties of this mortar drastically decreases at high temperature and attributed to the enhanced liquid phase formation at high temperature. Moreover the bonding strength of the sample more at high temperature due to the bonding of the bricks by the glassy phase formed at high temperature.

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